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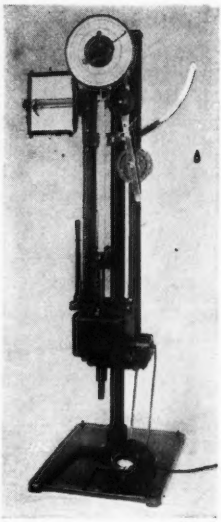

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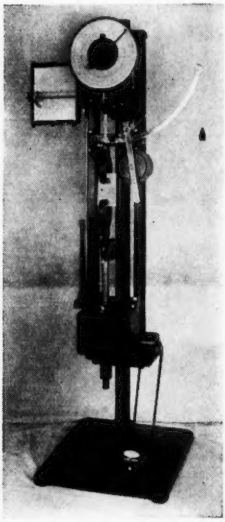

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Textile Materials and the Time Factor
I. Mechanical Behavior of Textile Fibers and Plastics

By HERBERT LEADERMAN *

A Textile Foundation Publication

Abstract

Some relations between stress, strain, and time in high molecular weight organic materials are discussed. In the light of these relations a better interpretation can be given to the conventional stress-strain test than is possible in terms of the mechanical properties of metals.

The properties of primary creep, secondary creep, swelling and thermo-recovery, and mechanical conditioning which are found in long-chain substances are discussed in detail, together with some results that have been obtained on silk and rayon.

Introduction

THE art of processing vegetable and animal fibers to make textile materials is of very ancient origin; very early in the story of civilization the peculiar properties of textile fibers were recognized and the techniques developed to make them serviceable to man. From this very old industry has arisen one of the newest—the art of making synthetic fibers which are either, as in the regenerated celluloses, partly synthetic, or as in the case of nylon and vinyon, entirely so. These synthetic fibers are in part replacing the natural fibers heretofore used.

Textile fibers, both natural and synthetic, are particularly suited for their various applications by virtue of their special properties, especially their mechanical properties. There is no doubt in the mind of the layman that the mechanical properties of textile fibers are quite different from those of, say, hard metals or chemical solutions. It is precisely because of this difference that the technician has difficulty in evaluating the significant mechanical properties of a textile material, be it for domestic or industrial use.

In attempting to describe the mechanical properties of textile materials, the problem has been approached from the viewpoint of either the engi-

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neer or the chemist. Neither of these lines of approach has been satisfactory; meanwhile, exactly the same problem has arisen in connection with the description of the mechanical properties of plastics and rubber. Much light can be thrown on the subject, however, by considering these materials as bodies which are on the whole elastic solids but in which time and other effects play an important part. It is to these effects that textile fibers must owe their valuable properties, and this article is concerned with a discussion on these effects.

Differences between Textiles and Engineering Metals

It would be possible for us to draw up a specification for a mechanical test on a textile material following engineering practice, in which, for example, a required ultimate strength and perhaps a required ultimate strain are to be attained. We know, of course, that there are many factors of minor importance in metals testing which have to be taken into account. The temperature and the relative humidity of the atmosphere before and during the test have to be specified; owing to the scattering of the results, these should be treated statistically. In an attempt to control the time factor we could, for example, apply and remove the load at a given rate, as in the inclined plane tester.[1] Under these conditions we should be able to obtain reproducible data.

We have now to interpret these data to obtain information concerning *desirable* properties of the material under test. As has already been said, since the desirable properties of a metal are different from the desirable properties of a textile fiber or plastic material, the results of a mechanical test will have to be interpreted differently. Our object therefore is first—to consider some of the properties of textile fibers which may be included under the heading of time effects, and how they reveal themselves in mechanical tests; and second—to attempt to derive from these properties some conclusions concerning the ultimate structure of textile fibers, rubber, and plastics. From these conclusions we might be able to predict in a general fashion the expected behavior of a new material, and the conditions necessary to obtain desired characteristics.

Perfect and Imperfect Elasticity

First of all it is necessary for us to have a clearly defined terminology. For example, what do we mean when we say that a material is perfectly elastic? A layman might say that rubber is more "elastic" than steel, or that a rayon filament is more "elastic" than a cotton fiber, when he means that the first mentioned of each pair is more *extensible*. An engineer would say that steel is more nearly perfectly elastic than rubber; he defines *perfect elasticity* as the ability of a material under stress to return immediately to its unloaded form on removal of stress. If on removal of load the material does not thereupon completely regain its original form, such a material he calls *imperfectly elastic*. The point where the extension ceases to be proportional to load the engineer refers to as the "proportional limit"; the point at which the material ceases to be perfectly elastic as defined above is called the "elastic limit." The limit of proportionality and the elastic limit are considered to be practically identical. The concepts embodied in the above definitions have to be greatly modified when we come to consider the mechanical behavior of textile fibers and plastics.

Creep and Creep Recovery

First let us consider the behavior under load of a steel specimen at elevated temperature. Immediately on application, say, of a tensile load there will be an elastic extension. If the load be maintained at a constant value, a gradual "flow" or "creep" will take place. When the load is removed, there will be an instantaneous elastic contraction corresponding to the previous extension; thus a permanent set, corresponding to the creep under load, will remain, the magnitude of which depends on the stress, the nature of the steel, the temperature, and the time under load. Steel at elevated temperatures is thus an example of an imperfectly elastic body.

Now let us suppose that we carry out a similar experiment, first performed over a hundred years ago, on a raw silk filament. If a constant tensile load be applied to such a filament under ordinary temperature and humidity conditions, there will be, first an immediate elastic extension on application of load, and then a gradual "flow" or "creep" as in the case of steel. As before when the load is removed, there is an instantaneous contraction corresponding to the original elastic extension, leaving the filament with a length somewhat greater than its original value. If now the filament (free from load) be allowed to rest undisturbed, a remarkable and unexpected phenomenon will occur. The filament will gradually contract in length, and, under suitable conditions, may even eventually contract back to its original length. We call this phenomenon *creep recovery.* This effect, first observed on a raw silk filament, was discovered by Weber in 1835.[2] Weber realized that when the specimen was able to recover its original length (or in other words, when the recovery was *complete*), the creep and the subsequent recovery were really two aspects of a single phenomenon, which we can think of as a delayed elasticity. Upon application of a constant load to a filament we have an instantaneous elastic deflection followed by a delayed elastic deflection or creep. On removal of load we have an instantaneous elastic contraction followed by a delayed contraction or creep recovery. Weber suggested as a name for this phenomenon "elastic after-effect" ("elastische Nachwirkung"). In the minds of many people this name suggests only the creep recovery and not the preceding creep; to avoid any such confusion we will call this phenomenon *primary creep.*

Primary and Secondary Creep

In the first example of a steel specimen under tension at an elevated temperature there will actually be a creep recovery after removal of load, but in practice the magnitude of this recovery is usually slight compared to the total creep. Comparing this case with the case of the raw silk filament, in which (under certain conditions) it is possible to obtain complete recovery, we see that basically there are two types of creep phenomena. In the silk specimen all the creep was eventually recovered, hence the prenomenon of primary creep only was present. In the case of the steel, corresponding to the slight creep recovery there must have been an equal creep deflection of the nature of primary creep. Now we can imagine that superimposed upon this was the much larger non-recoverable creep component, which we may call, in general, *secondary creep.*

Plastic and Viscous Flow

The nature of primary creep is such that the creep rate for constant load decreases markedly with time and that eventually creep ceases. In the case of many materials which show creep under load, e.g., steel and glass at elevated temperatures, asphalt, etc., the creep rate for constant load decreases to a given value and then maintains itself at that value. The uniform flow corresponds to a *particular case* of the superimposed secondary creep which is called *plastic flow* or *viscous flow* according to the relation between flow rate and stress. In the materials we are going to consider, however, secondary creep usually appears to follow more complex laws than in the case of glass and steel.

It is interesting at this point to draw a distinction between solid and liquid materials. If a material flows under stress yet shows evidence of creep recovery, we must consider it to be at least to some extent a solid. Such materials are those considered above. If on the other hand, no matter how viscous they are, they show no evidence of creep recovery, we must consider them as liquids. Such a material is molasses.

Creep and Textile Materials

If we were to carry out a creep test under constant load on the raw silk filament discussed above, we should find that although the rate of creep decreased rapidly with time, it would require a length of time of the order of 24 hours before creep ceased. We would then find that the original extension had been about doubled, or in other words the magnitude of the delayed elastic extension was of the same order as the magnitude of the immediate elastic extension. In the case of glass at room temperature (when viscous flow is negligible) the total delayed extension is about 3% of the immediate extension. For a period of some fifty years glass was the only material to be studied extensively. Although much definite information was obtained concerning primary creep, the relative smallness of the effect in glass and the soft metal wires studied suggested that the phenomenon might be of minor importance. In recent years, however, much work that has been carried out on textile fibers such as rayon, wool and silk, on plastics and on rubber shows that in all these materials there is a very marked primary creep. Consequently, interest in the problem of primary creep has been revived, especially as it seems that the bulk of the experimental and theoretical methods previously developed is applicable with but slight modification to these materials.

Complete Elasticity

We can see that, when primary creep is present in a solid, even though the deflection may be proportional to load immediately after application of a constant load, this will not be the case sometime later, since the deflection would depend on the time that has elapsed since the load was applied. Also, in general, we should expect the deflection to be different if the load were applied gradually instead of instantaneously. If, following removal of load, the resulting increase in length was eventually completely recovered, we could not think of our material as being imperfectly elastic. We can, however, introduce a new term to describe the behavior of such materials.

If within certain load limits, the creep is such that it is entirely recovered subsequent to removal of load, we may call such a material *completely elastic.* Just as the elastic range in a metal may be modified by suitable mechanical treatment, it seems also that we can modify the conditions under which a material is completely elastic. This preliminary treatment we can think of as *mechanical conditioning.*

In the Textile Laboratory of the Massachusetts Institute of Technology two research projects are being carried out on the mechanical properties of filamentous materials. One, sponsored by the Textile Foundation, is concerned with primary creep; the other, which is concerned with the stress-strain relations in silk filaments, is sponsored by the International Silk Guild.* In both these investigations it is necessary to "condition mechanically" the specimens in order, in the first case, to study primary creep without the complicating factor of secondary creep, and in the second case to study the stress-strain behavior under conditions of complete elasticity.

As a result of these investigations, the conditions necessary to obtain complete elasticity can be tentatively stated. It appears that a fiber can be "mechanically conditioned" up to a given load by subjecting that fiber to this load for a long period of time (say 24 hours); provided, of course, the load is not too great. Under these conditions, there will be a primary (recoverable) creep, and superimposed upon this a non-recoverable secondary creep. After removal of load and allowing to recover, there will be a permanent set[3] on repeating the experiment at the same or lower load, however the fiber should be now completely elastic. Alternatively, it seems that the same result can be obtained by gradually loading the fiber to the given load, and then graually removing the load. If this process is repeated several times, the specimen will be put into a completely elastic condition.

Thermo- and Swelling Recovery

The concept of conditioning a specimen mechanically to obtain a desired elastic behavior is not an unexpected one for engineers, who are familiar with the phenomenon of "work-hardening." Engineers are also familiar with the fact that the hardening effect of cold-work can be removed by heat-treatment. In the case of textile materials and plastics, however, not only can the "hardening" effects of "permanent" strain be partly or wholly removed, but also the strain itself.

As an example, if raw rubber be stretched and then allowed to recover, it is found when recovery finally ceases that there is a permanent set. If now the specimen be heated to say 100° C. for a short length of time, and then cooled back to room temperature, it will be found that the permanent set will have disappeared to a large degree.[3] This phenomenon has been called *thermorecovery.*[4]

If on the other hand we take say a rayon yarn in the dry condition, we would expect this filament to swell longitudinally on immersion in water, and to contract back to its original length on drying out. It has been demonstrated, however, that if the yarn had been previously stretched slightly in the dry condition, the swelling on immersion in water is less than would be expected, and might even be negative, i.e., on wetting-out a contraction occurs. In this way a large part of the apparently permanent set

* The writer wishes to acknowledge the kindness of the International Silk Guild and Mr. H. Hindman in allowing him to quote some of their results.

is regained on drying.[5] This phenomenon we might call *swelling recovery.* In connection with the two programs of research referred to above, this property is currently being studied in single filaments of silk and rayon. As a further example of this phenomenon, if the stretched raw rubber in the case above be subsequently swollen in benzene vapor or a benzene-alcohol mixture and then dried, there will be a further contraction.[6] This again would be a swelling recovery.

"Freezing-In" of Strain

A final property we shall consider will be that known as the "freezing-in" of strain. It is not a basically new property, since it can be explained in terms of the primary creep properties of a substance; for this reason we can consider it as a kind of time-effect. This property is manifested by many materials which show a marked primary creep, and will be illustrated by two examples. One is the application of this phenomenon to the problem of three-dimensional photoelasticity. A bakelite beam under a constant bending moment if heated to about 110° C.—120° C. will assume very rapidly a large but definite end deflection.[7, 8] If the loading be removed, there will be a rapid complete recovery. If on the other hand the specimen be cooled down to room temperature while carrying the load, and then the load be removed, most of the deflection is preserved apparently indefinitely. The specimen can then be sawn up and the strains in the interior examined. The term "freezing-in of strains" is thus seen to be a very apt one. If, however, after the removal of load at room temperature the specimen be reheated, the locked-in strains will disappear.* Exactly the same happens in the case of rubber, except that in this case the so-called transition temperature lies below room temperature; that is, the specimen has to be cooled after application of load at room temperature. Furthermore, through the property of primary creep the behavior of rubber at low temperatures can be explained. The elucidation of these phenomena must be deferred, however, until we first consider in detail some of the primary creep properties of high molecular weight organic solids.

Primary Creep and the Memory Effect

So far we have considered the various aspects of the behavior of textile fibers and other high molecular weight substances under load. Of these properties that of primary creep can usually be separated from the others and studied independently; for this reason, this was the first property to be studied in detail. As we have already mentioned, the primary creep properties of these materials follow with but slight modification the same properties of glass and soft metals. We can, therefore, review with profit some of the early work on primary creep, in order to understand how our present ideas have developed.

We know that if we apply a constant load to a specimen, and that if only primary creep is present, the creep will proceed at a gradually decreasing rate. If after some time the load be quickly removed, the specimen shows a remaining distortion which, however, gradually disappears but also at a decreasing rate with time. This, of course, is the aspect of

* The release of "frozen-in" strain by reheating or the acceleration of creep recovery by heating must not be confused with thermorecovery.

the phenomenon which we call creep recovery. It was found that for reasonably short durations of time under load the time for recovery was very long. The early workers set themselves the problem of elucidating the nature of the creep recovery by applying a constant stress or strain for various lengths of time.[9] Though, for example, in a tension test the length of the specimen in the recovery period approaches the natural length only asymptotically, we can arbitrarily assign a recovery time to that necessary for recovery to a certain position. We would then find that the shorter time under load requires a relatively longer recovery time. For example, if a specimen be allowed to creep for one minute, it might require some ten minutes to recover; while if it be allowed to creep for one hour, it might require *four* times this length of time; if the specimen be allowed to creep for 24 hours, the recovery might require about an *equal* length of time. In contrast to the complex effect of the variation of time under load, the effect of the variation of load, keeping the creep time constant, seems to be much simpler. In glass, bakelite,[10] and some other materials the effect of increase of the load used seems to be to increase all the deflections, both in the creep and recovery periods, in the ratio of the increase in loading weight.

To explain these results Boltzmann [11] (in 1874) advanced an hypothesis, which he called the "Superposition Principle," and which can be stated as follows. *At any instant, the deflection of a solid manifesting only primary creep can be separated into two components. One component is the instantaneous elastic deflection, proportional to the load acting at that instant. The other component is the primary creep or delayed elastic deflection dependent not only on the load acting at that instant but also on the entire previous loading history. Hence as far as the primary creep component of the deflection is concerned, the material possesses a "memory" of all past loading actions.* On this account this hypothesis is sometimes referred to as the "Memory Law" of creep.

Boltzmann expressed the memory action hypothesis in a mathematical form; this is given in a somewhat simplified presentation in the appendix. For the case when the load is increased or decreased in steps (as distinct from gradual load changes) the creep and recovery can be predicted by very simple rules.

At first sight the postulate of a memory action seems somewhat arbitrary; indeed it was not generally understood or accepted [12, 13] by Boltzmann's contemporaries. Therefore, before discussing the conclusions that can be drawn from the mathematical theory, a famous experiment of the physicist F. Kohlrausch [14] will be described. Kohlrausch had been actively interested in creep research for many years. It occurred to him—a point which seemed to have been missed by Boltzmann—that if a material possessed a memory, then by suitable choice of loading history, the creep recovery could change its direction of motion. For example, suppose that a specimen—say a rubber thread in torsion—be loaded, first for a long time in one sense, and then for a short time in the opposite sense. Then, if the loads and loading durations are properly chosen, it is possible that immediately after release of load the "memory" of the second action might outweigh the memory of the first loading action, and the direction of creep recovery will be in the opposite direction to that of the later load, even if this necessitates the specimen recovering *away* from the equilibrium position. After some time, however, the memory of the first loading action will outweigh the memory of the second one and the direction of creep recovery will then

change, the specimen recovering towards the equilibrium position in the usual way. In an exactly similar way, if this memory action exists, it would be possible to cause the direction of creep recovery to change *twice* by suitably changing the direction of loading twice.

Now let us consider some of the results which Kohlrausch obtained, following the above schemes, by loading a rubber thread in torsion and tension and a hard rubber beam in bending and then observing the creep recovery. These results, as given by Kohlrausch, are reproduced in Fig. 1. The Recovery Curves I, II, and III each show a change in direction. Curve

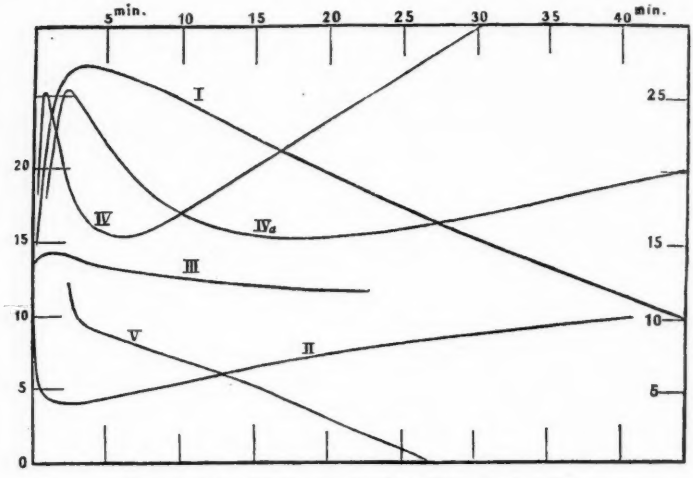

Fig. 1. The Memory Effect (Kohlrausch's Results).

V, for which the loading conditions were not properly chosen, shows merely points of inflection. Curve IV, redrawn with three-fold abscissa as Curve IVa is the most interesting. Since the direction of the recovery changed twice, the deflection from the final position was actually *increasing* for a period of time. Considering these results, one is drawn to the conclusion that primary creep behaves as if the specimen remembers the previous loading actions.

Granting now that some sort of memory action as postulated by the Superposition Principle exists, we now went to consider what are the quantitative predictions of the Principle for some simple loading "histories," and to see to what extent these predictions are confirmed by actual test.

The Superposition Principle: Creep and Recovery

The creep properties of textile fibers manifest themselves in several ways. The most convenient experimental method of studying these properties is the observation of the longitudinal strain creep of single filaments. A constant load is applied to a filament as rapidly as possible, and the

extension is measured from time to time by means of a cathetometer. Similarly, the contraction is measured from time to time following removal of load. We will assume that even if the length of time under load be 24 or 48 hours, following removal of load the specimen will eventually contract back to its original length; i.e., we assume that the specimen has been previously "conditioned" so that secondary creep is absent.

Such a creep test under constant load we may call a *long-duration creep test.* From this test, if secondary creep is absent, we can, in general, predict the creep and recovery behavior for any loading history in which this load is applied or removed at any arbitrary times. If the behavior as predicted by the Superposition Principle agrees with the observed behavior, then we can say that the Principle gives a good representation of the phenomenon of primary creep. Experiments that have been carried out in the Textile Laboratory of M. I. T. on single filaments of viscose, nylon, and silk, and also on collagen, indicate that this is so.

The Superposition Principle can be tested most simply by a creep and recovery test, in which the load is removed after a specified time and both the creep and the subsequent recovery observed. For the recovery deflections, the Boltzmann theory reduces to the following simple rule: *We assume that the effect of the original load, as far as its creep effect is concerned, to continue indefinitely. Superimposed upon this we assume a (negative) creep due to an equal negative load applied at the instant of load removal.*

Suppose we have carried out a long duration creep test, at a given load. We should like to predict what the creep recovery would have been if the load had been removed after, say, ten minutes had elapsed. The creep

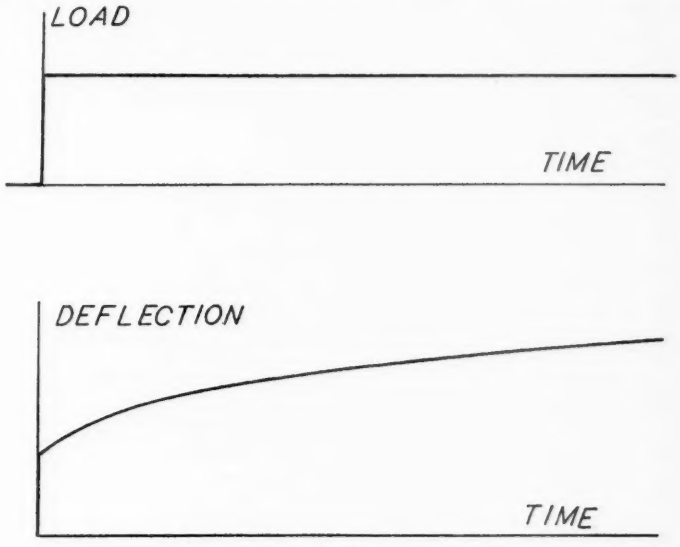

Fig. 2.

recovery five minutes after load removal, from the Superposition Principle, would then be merely the difference between the deflection after 15 minutes in the long-duration creep test and the deflection after 5 minutes. This case is illustrated graphically in Figs. 2 and 3. In Fig. 2 are shown the load/time and deflection/time diagrams for a long-duration creep test. Fig. 3 shows first, the load/time diagram in which the load is removed at t_1 minutes after the instant of application; then the equivalent loading diagram in which a positive load applied at zero time continues indefinitely, and a negative load, applied at time t_1, also continues indefinitely. In the lowest diagram Curve X represents the long-duration creep curve due to the positive load applied at zero time, and Curve Y the long duration creep curve due to the negative load applied at time t_1. The deflection at any time t in the recovery period should then be merely the corresponding vertical distance between the curves X and Y. If this rule is true we can see that in a general way the recovery time is longer than the time under load, and that the time for recovery is relatively longer, the shorter the time under load.

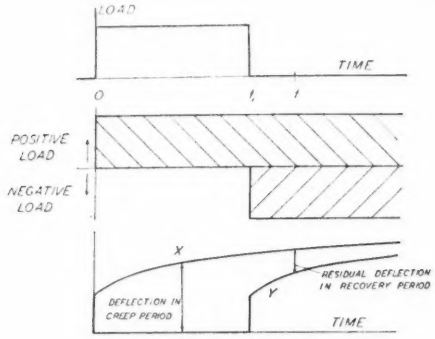

Fig. 3.

Recovery after Long Duration Creep

Now let us see what the Superposition Principle predicts for the creep recovery following a long duration creep test. By way of a concrete example, let us again suppose that we have carried out a creep test of 24 hours' duration under constant load on a silk or rayon filament; of course, we assume that only primary creep is present. Let us assume that the creep had reached its limiting value, L (Fig. 4). Then applying the above rule, it is seen that the course of the recovery should be the "inverse" or "mirror-image" of the course of the creep. Corresponding to the initial instantaneous extension on application of load we have an equal instantaneous contraction on removal of load; corresponding to the initial rapid extension we have an initial rapid contraction, and finally, recovery is complete in the same time as that required for the creep to reach completion. If, however, the creep had not quite reached its limiting value at the time the load was removed, then according to the Superposition Principle we would expect the recovery to proceed somewhat more slowly in the final

stages than the corresponding creep. In actual practice when the creep or recovery become very slow, slight variations in temperature or humidity cause an appreciable disturbance, so that a check of the Principle in this region is not possible.

Now it has already been mentioned that the rate of creep for primary creep under constant load decreases at first very rapidly with time, but subsequently takes a long time to reach completion. We can accordingly obtain a better idea of the process by plotting to a logarithmic time base. The change in length of the filament can be measured for example by a

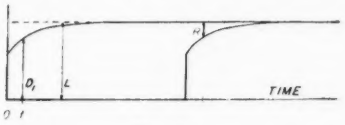

Fig. 4a.

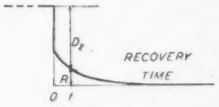

Fig. 4b.

cathetometer. Then the extension at any time during the long-duration creep test is simply the difference between the reading at that instant and the reading of the length before the application of load. Similarly, the contraction during the subsequent recovery can be measured. If we, therefore, plot the *residual extension* (R) in Fig. 4 for the recovery following long duration creep against recovery time, we should obtain a curve which is the mirror-image of the curve of extension (D_1) against time for the long-duration creep test. We could test this easily by plotting the residual extensions in the recovery with the ordinate scale inverted; better still, we could compare the *contraction* (D_2) in the recovery stage due to removal of load with the *extension* (D_1) in the creep stage due to application of load. From Fig. 4(b) we see that for corresponding creep and recovery times these deflections should be equal.

In Fig. 5 are plotted the extension during a long-duration creep test as a function of time (to a logarithmic scale) and also the subsequent contraction due to removal of load. The extensions and contractions are expressed as percentages of the initial length; the material was a silk filament under a load of 0.53 gm. It will be observed that over a period of 24 hours the deflection is increased by one-half of its initial value. It will also be observed that the creep and recovery observations differ by a small amount due to the initial elastic extension being slightly greater than the initial elastic contraction. This effect is small; it cannot be said at present whether this is due to faulty experimental technique or whether it is a real effect.

The Superposition Test

It has already been explained how the course of the recovery can be predicted from the creep curve under constant load by means of the Superposition Principle. By an extension of the rule given above, the Principle can be used to predict the creep following reapplication of the same load, and the subsequent recovery. For those materials in which, as assumed by Boltzmann's theory, both the creep and the instantaneous deflections are proportional to load, the rule previously given can be extended to cover all values of load within the limits of complete elasticity. The extended rule

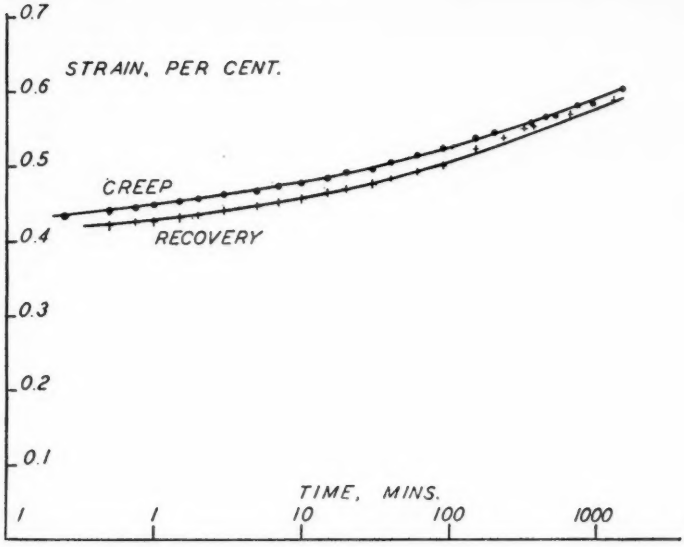

Fig. 5.

is then as follows: *assume all increases in load to be equivalent to the application of a positive load, equal in magnitude to the load increase, at the instant of load increase. Similarly, decrease in load is assumed to be equivalent to the application of a corresponding negative load.*

As before, the effect of each load, positive or negative, is assumed to continue indefinitely, and the deflection at any time is simply the sum of the deflections due to each separate loading action. Each of these separate effects can be obtained from the long-duration creep test.

By way of example, assume a given load is applied at zero time, removed at time t_1, reapplied at time t_2, and subsequently removed at time t_3. The loading and equivalent loading diagrams are shown in Fig. 6. The uppermost deflection curve is the long-duration creep curve for a load applied at zero time. The subsequent curves are drawn to commence at times t_1, t_2 and t_3. Then the deflections at the time t due to the positive load applications at zero time and time t_2 are $+ W$ and $+ Y$. The deflections due to the negative load applications at times t_1 and t_3 are similarly $- X$ and $- Z$. Hence the deflection at time t due to the four separate loading effects is $W - X + Y - Z$. These deflections correspond to deflections in the long-duration test at times t, $(t - t_1)$, $(t - t_2)$ and $(t - t_3)$ respectively.

We thus have a second powerful means of checking the Superposition Principle. To return to the previous example, we can measure the extension during a loading period of, say, ten minutes, which we can call the first creep period, we can then remove the load, and measure the extension in the first recovery period, also of ten minutes' duration. We can then reapply the load for a further 10 minutes, and thus measure the deflections

in a second creep and a subsequent second recovery period each of 10 minutes' duration. By means of the Superposition Principle, we can compute from a long-duration creep test the extensions to be expected. Al-

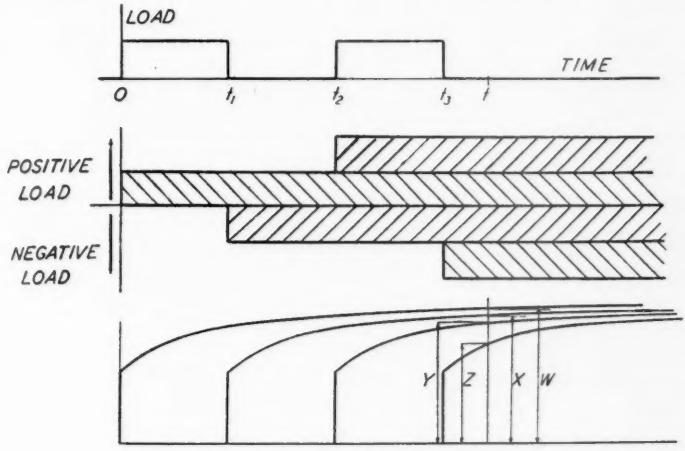

Fig. 6.

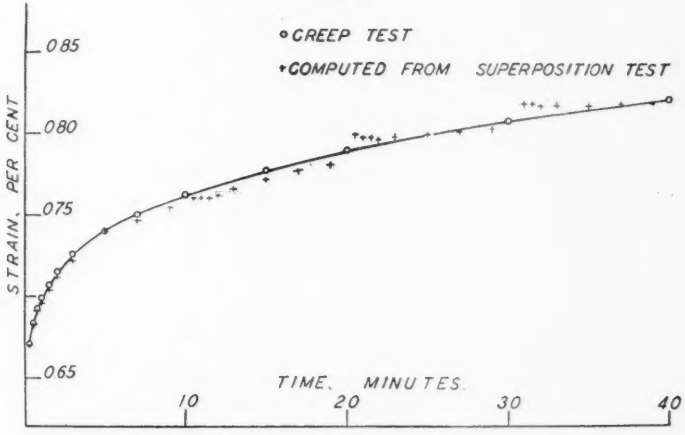

Fig. 7.

ternatively, we can compute the long-duration creep extensions from the superposition test data.

In Fig. 7 the latter has been done. The circles represent the longitudinal strains plotted against the corresponding values of time for the

first forty minutes of a creep test on the same silk filament as above, but under a load of 0.86 gm. In the superposition test the specimen was under the same load from zero time to 10 minutes, and from 20 to 30 minutes; in the other periods the specimen was free from load. The crosses represent the values of strain for the creep curve under constant load computed from the superposition test data; the discrepancies can be fully accounted for by the difficulty in applying or removing the load at exactly the right instant, and the vibration set up in applying or removing the load. Viscose and nylon show exactly the same behavior, and it is to be concluded that in these materials the creep and recovery due to the application or removal of a given load can be satisfactorily explained by the Superposition Principle.

The Creep Curve for Constant Load

It seems that the fundamental test in the study of primary creep is the long-duration creep test under constant load. From this we can derive the recovery curve and the data for the superposition test. Furthermore, by studying the plots of the creep curve against the logarithm of creep time, we can investigate the effect of the magnitude of load on primary creep.

The general nature of the deflection under constant load, when plotted against the logarithm of the creep time, is as shown in Fig. 8. The deflection curve is sigmoidal, with a lower horizontal asymptote representing the instantaneous elastic deflection, and an upper horizontal asymptote representing the final deflection. It may happen that the left-hand portion of the curve may represent times which are too small to allow recording of any observations. It may also happen that the right-hand portion of the curve represents times which are so long that it is impossible in practice to investigate this region. Let A be the instantaneous deflection, represented by the lower asymptote, and let B be the creep component of the deflection at any convenient time, say, 60 minutes after application of load. Then the total deflection at any time t can be represented by

$$Y_t = A + B\psi(t) \qquad (1)$$

where $\psi(t)$ is a function of time, and is the ratio of the creep component at time t to the creep component, in this case, at 60 minutes. Alternatively, if the material is such that the upper asymptote can be reached in a reasonable time, we might make B equal to the total creep deflection, and in this case $\psi(t)$ represents the ratio of the creep component of the deflection at time t to the total delayed deflection. The function $\psi(t)$, which we call the *creep function*, is then said to be "normalized."

The effect of different loads can then be found by conducting long-duration creep and recovery tests for different loads and comparing the values of A and B obtained. It seems that for the various kinds of rayon filaments tested the instantaneous elastic deflection A is very nearly proportional to the load. The same seems to be true for bakelite [10] (as used for photoelasticity tests) and for both hard and soft rubber.[15]

The original Boltzmann theory postulated that the delayed component of the deflection as represented by B should also be proportional to load, and this seems to be true for glass [13] and bakelite.[10, 16] It has also been reported to be true for the hard and soft rubber [15] quoted above. On the contrary, H. de Witt Smith [17] found that this was no true for viscose; this result has been confirmed by the writer, who has found the same to hold

for nylon and silk; this also has been found to hold for collagen. It seems possible in all long-chain polymers with no cross-linking that this deviation exists, whereas in cross-linked material such as vulcanized rubber and bakelite the creep is proportional to stress.

We can investigate the relation between the magnitude of the creep and the load in the following way. First, by extrapolating the creep curve

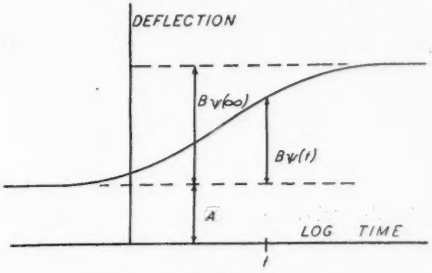

Fig. 8.

under constant load to zero time we can separate the instantaneous elastic deflection from the delayed or creep component. The former is proportional to load; even if the latter is not proportional to load, it can be shown that for different loads the curves obtained are similar in shape, and differ only by a scale factor. We wish to determine the relation between this

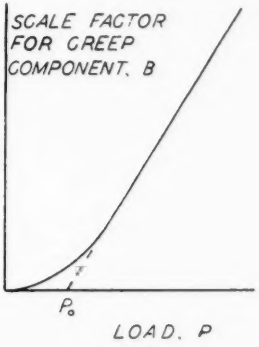

Fig. 9.

factor, which corresponds to B in equation (1), and the load. This necessitates carrying out a series of long-duration creep and recovery tests at various loads under very carefully controlled conditions of relative humidity and temperature. The results show roughly that the magnitude of the creep increases linearly with load, that is, the plot of the scale factor of creep against load can be represented by a straight line which intersects the load axis at some load P_o. As indicated in Fig. 9, it seems probable that

the foot of the curve departs from the straight line to bend round to the origin; though in this region the creep is too small to measure accurately and to be of importance. Thus such long-chain materials should behave for higher loads as if they have a "creep limit" P_o. The creep is then proportional to the excess of the load over this limiting load, i.e. to $(P - P_o)$.

If we, therefore, run a series of long-duration creep and recovery tests for various loads on a long-chain polymer, then the instantaneous elastic deflections plotted against load should give a straight line, the slope of which represents the "instantaneous" or "acoustic" modulus of elasticity.[15] The delayed components of the creep or recovery deflection divided by the appropriate value of $(P - P_o)$ and plotted against the logarithm of creep- or recovery-time should yield a unique curve, from which the creep behavior of the material under any loading history at loads not greater than the "conditioning" load can be predicted. Such curves have in fact been obtained with viscose and nylon, and it is felt that the creep of other chemically similar materials should follow a similar behavior.

Behavior of a Material under Cyclic Loading.

In practice, if it were desired to obtain information of the mechanical behavior of a textile material, say, a rayon yarn or a silk filament, the specimen would be placed in a testing machine to obtain a stress-strain curve. The testing machine should be one in which the load is applied and removed at a constant rate, as in the inclined plane and chainomatic arrangements. Because of the simultaneous occurrence of a Hooke's Law elasticity and primary creep, together with the possibility of conditioning and secondary creep being present, the interpretation of load-extension curves thus obtained would be expected to be very complicated. The results * of cyclic loading tests on silk filaments obtained in the Textile Laboratory of the Massachusetts Institute of Technology are consistent with the creep behavior outlined above.

First, let us assume that the filament under test is already mechanically conditioned up to the maximum load to be used in the test. On applying the load at a given rate, the load-extension curve will not be linear, due to the effect of primary creep, but will be concave towards the extension axis. If when a given load is reached the load be then reduced at the same rate as before, then, due to the fact explained above that the recovery is always slower than the creep, the extension at any given load on the load-decreasing path is greater than at the same load for the load-increasing path, as shown in Fig. 10. Finally, when zero load is reached, the length of the specimen is greater than its original value. According to the conventional engineering interpretation, the proportional limit is very small, the elastic limit has been exceeded, and a hysteresis loop has been formed, finally leaving a permanent set. We know, however, that, given sufficient time, the apparent "permanent set" will disappear, and the specimen will recover to its original length. The apparently imperfect behavior of the silk filament is actually attributable to the desirable property of primary creep. Since the filament is in exactly the same condition as in the beginning of the test, this cycle can be repeated at will; such has indeed been found to be true.

Tests were then carried out on the same filament at half the rate of loading, and at the same rate of loading but to only half the maximum

* Research sponsored by the International Silk Guild.

load. In the first case, since the time for creep and recovery was increased, the effect was roughly to shear over the load-extension loop along the extension axis. In the second case, the loop width was very small—very much less than half the loop-width at full load. If the creep were proportional to load, as in bakelite, we would expect the loop width to be halved. The results are, therefore, consistent with the non-proportionality of creep and load.

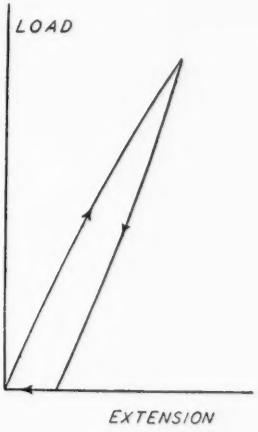

Fig. 10.

Finally, consider mechanical conditioning. This has been achieved as already described, by loading the silk filament for several cycles. After the first cycle, recovery was only partly complete. After the third or fourth, the fiber contracted back to its length at the beginning of the cycle. In this case, too, the apparent permanent set could be removed entirely or in part by treatment with water or steam. Thus we see that the complex load-extension behavior of silk filaments under cyclic loading can be explained, not in terms of the engineer's concepts of mechanical behavior, but in terms of the general primary creep behavior of long-chain polymers and of the mechanical conditioning and swelling recovery properties of these materials.

Effect of Temperature on Primary Creep

The phenomenon of freezing in of strain has been described earlier; it was stated that this was really an aspect of primary creep. M. Hetenyi * has measured the creep deflection of bakelite beams under a given bending moment at various temperatures. His results are shown in Fig. 11. It will be noted that while the final creep deflection was the same for all the temperatures of test, the time taken to reach the final deflection varied enormously with temperature. It was mentioned previously that we are

* The writer wishes to express his appreciation to Mr. Hetenyi for kindly supplying the data of the above tests.

dealing here with what is a primary creep phenomenon, since removal of load resulted in a complete creep recovery. If now the creep curves under constant load be replotted against the logarithm of the creep time as in Fig. 12, we see that we have essentially the sigmoidal curve characteristic of primary creep. The effect of change of temperature appears to displace this curve along the axis of logarithmic time (i.e. to extend or contract the time scale). It will be noted that at the higher temperatures only the upper part lies in the observable range of time, while for the lower temperatures only the lower part can be measured.

According to Hetenyi, whereas at 120° C. the final deflection was reached in about one or two seconds, about ten thousand years would be required to reach the limiting deflection at room temperature. Thus if the

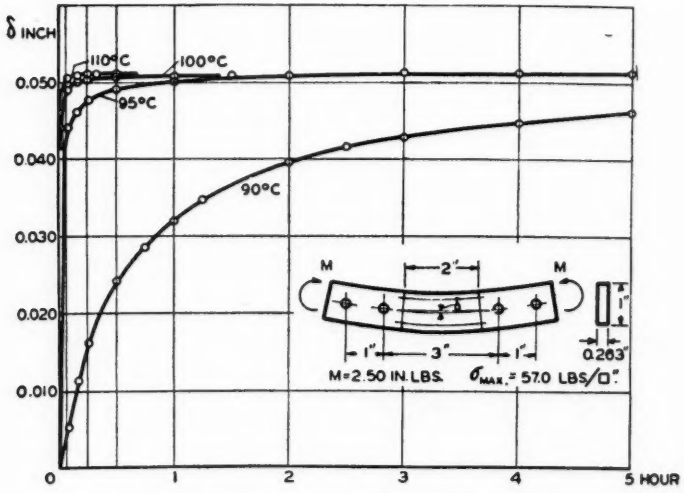

Fig. 11. Creep of Bakelite at Elevated Temperatures under Constant Load (Hetenyi's Results).

specimen be heated to 120° C., loaded, cooled, and then unloaded, the time scale for the recovery would be so extended that in practice the strain can be considered as permanently "frozen in." Consideration of the differences in the sigmoidal creep curve for different materials, and the quantitative explanation of the temperature effect will be considered in the second part of this article. It should be pointed out, however, that while the creep of bakelite in the example above is very small at room temperature compared to that at 120° C. (owing to the very extended time scale in the former case) the total creep deflection in both cases should be the same. The point is of interest as to whether in textiles the effect of change of temperature and of humidity is of the same nature as the effect of temperature on the primary creep in bakelite: that is, to cause an extension or contraction of the time scale.

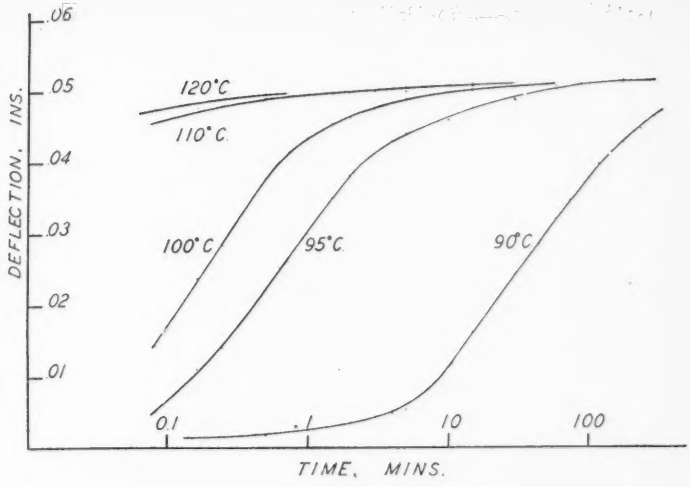

Fig. 12.

Primary Creep in Rubber and Hard Rubber

It is well known that if rubber is cooled down to the temperature of liquid air, it undergoes a transformation in properties; it becomes brittle and glass-like. On the other hand, hard rubber heated to 150° C. shows properties similar to those of rubber at room temperature. It has been considered that the change in the properties of rubber were due to a transition taking place more or less at a definite temperature. Kobeko [15] and his coworkers, however, have shown that these changes can be explained simply in terms of the effect of temperature on the primary creep behavior of these materials. Figs. 13 and 14 show the creep recovery, after long-duration creep, of cylinders of rubber and hard rubber respectively, under constant torque. The deflections are expressed as shear strain per unit stress, and are plotted to a logarithmic time basis. The effect of variation of temperature is thus seen to cause a shift of the sigmoidal creep curve along the axis of logarithmic time. At very low temperatures the creep of rubber is insignificant; the deflection is merely the initial elastic deflection. This corresponds to an initial or acoustic modulus which is of the order of that of silk or nylon. The large reversible deflection or so-called "high elasticity" at room temperatures is due, therefore, to the existence of a primary creep which is very much greater than the initial elastic deflection. We see, therefore, that the mechanical behavior of rubber and hard rubber can be explained by means of their primary creep properties.

Other Applications of the Theory of Primary Creep

Only shortly after Boltzmann's theory was published, it was found that the dielectric of a glass condenser possessed a "memory" in its electrical properties. A condenser charged first in one direction, then in the

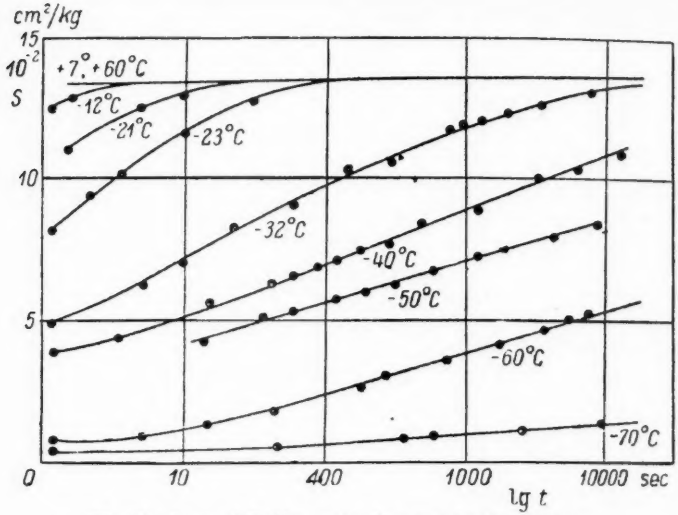

Fig. 13. Creep of Rubber under Constant Load (Kobeko).

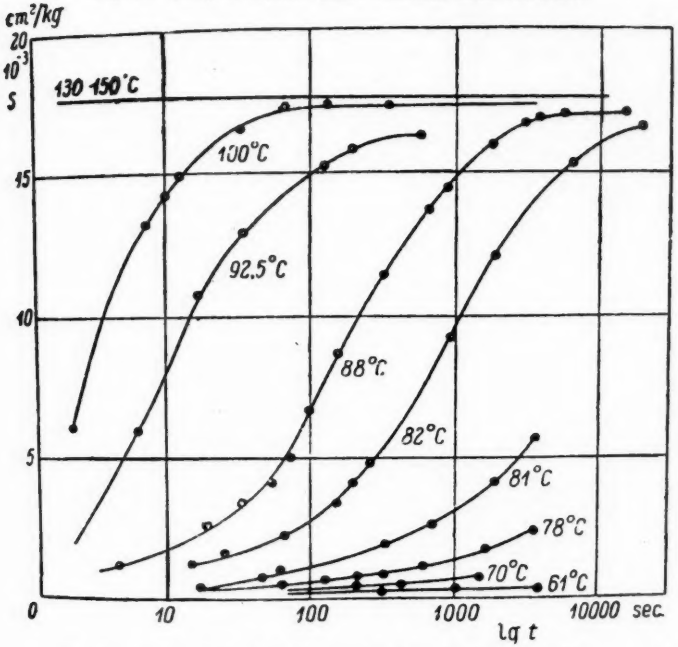

Fig. 14. Creep of Hard Rubber under Constant Load (Kobeko).

other, grounded, and then insulated, was found to develop a charge which under certain circumstances could reverse its sign.[18] The analogy is made clear if we consider for example a rubber thread which is allowed to creep under load. This is analogous to the charging of a condenser in which the charging potential corresponds to the constant load, and the charge corresponds to deflection. Removal of load corresponds to grounding the condenser. If now the creep recovery be interrupted by holding the specimen at a constant length, a gradual tension will be built up. This corresponds to the growth of the residual charge. The reversal of residual charge, corresponding to the reversal of creep recovery of Kohlrausch's famous experiment, was first demonstrated by Hopkinson. Maxwell suggested to him[19] that Boltzmann's theory could be applied to this problem. Today it is generally accepted that the dielectric of a condenser obeys the Superposition Principle, but the credit is usually given to Hopkinson and not to Boltzmann.[20]

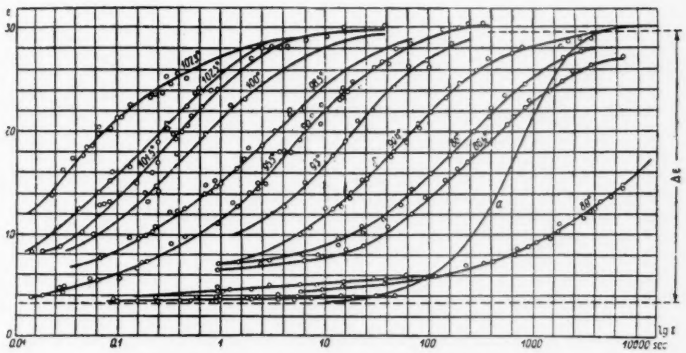

Fig. 15. Dielectric Creep in Phenolphthalein.

The analogy is completed by considering the discharge of a condenser, charged at constant potential for a long time. Fig. 15 represents the charge given up (corresponding to creep recovery) against time to a logarithmic scale for different temperatures for a condenser with phenolphthalein as a dielectric.[21] When the experimental difficulties involved are considered, there can be no argument but that the effect of temperature is simply a shift of the curve along the logarithmic axis of time. The analogy between electrical and mechanical creep is thus complete. At this stage space does not permit mention of the close analogy that has been shown to exist between the magnetic and mechanical creep of iron,[22] or mention of optical creep. It does seem, however, that studies of the optical and mechanical creep in textile fibers, and of the dielectric and mechanical creep in plastics, are bound to be of great interest.

We can now see that the behavior of plastic materials under load can be reduced to a simple pattern. The phenomena present are: the instantaneous elastic deflection and the primary creep, recoverable on removal of load; secondary creep, which corresponds to the viscous flow in glass-like materials and mechanical conditioning in long-chain polymers; and finally, thermo- and swelling-recovery, which represent the partial removal of the secondary creep.

The primary creep under any given loading history can be deduced from the creep curve under constant load by means of the Superposition Principle. The effects of change of load and of temperature have been considered. In a later paper of this series there will be explained how the remarkable creep properties can arise, the significance of the nature of the creep curve for constant load and the effect of temperature on this curve. The final task is then to show that the accepted picture of the ultimate structure of these long-chain polymers as deduced from chemical and X-ray evidence [23] is consistent with the mechanical behavior of such materials as outlined above.

Appendix to Part I

The General Superposition Principle

The Superposition Principle as enunciated by Boltzmann was concerned with stress relaxation following a given strain history. The separate relaxations of dilatational and distortional stresses was considered. Following Becker [24] and others, this separation is usually neglected, and the theory given below is developed in terms of the strain following a given loading history.

If Y_t is the deflection at time t and if P_t be the load at time t and P_θ the load at a previous time P_θ, then the elastic component of the deflection at time t is given by FP_t/E where E is the appropriate modulus and F a form factor. The creep component of the deflection at time t is due to the entire previous loading history. The creep deflection at time t due to a load P_θ acting at a previous time θ for a length of timd $d\theta$ is assumed to be proportional to $P_\theta \cdot \phi(t-\theta) \cdot d\theta$ where $\phi(t-\theta)$ is some function of the "elapsed time" $(t-\theta)$. It is assumed further that the creep deflection at time t is the simple summation of these effects due to the past loading history, then

$$Y_t = \frac{F}{E}\left\{ P_t + \beta \int_{-\infty}^{t} P_\theta \phi(t-\theta) d\theta \right\} \tag{2}$$

Introducing the elapsed time $\omega = t - \theta$, we get:

$$Y_t = \frac{F}{E}\left\{ P_t + \beta \int_{0}^{\infty} P_{t-\omega} \phi(\omega) d\omega \right\} \tag{3}$$

This is the general superposition equation, for the case of the creep proportional to the load.

For constant load P applied at zero time, this reduces to:

$$Y_t = \frac{F}{E} P \left\{ 1 + \beta \int_{0}^{t} \phi(\omega) d\omega \right\} \tag{4}$$

Replacing the integral by the function $\psi(t)$, this may be written:

$$Y_t = \frac{F}{E} P\{1 + \beta\psi(t)\} \tag{5}$$

For creep recovery after removal of load at time t_1, we get from equation (3):

$$Y_t = \beta \frac{F}{E} P\{\psi(t) - \psi(t - t_1)\} \tag{6}$$

This equation is seen to be consistent with the rule given previously for the

computation of creep recovery from the long-duration creep test. In a similar way, for creep recovery after a creep for a very long time t_1, we have

$$Y_t = \frac{\beta F}{E} P\{\psi(t + t_1) - \psi(t)\} \tag{7}$$

Since the first term is nearly constant, this is nearly the same as:

$$Y_t = \frac{\beta F}{E} P\{\psi(t_1) - \psi(t)\} \tag{8}$$

hence the recovery curve after long-duration creep is the mirror image of the creep curve.

Expressions for superposition tests can similarly be worked out. The above general equation applies to materials such as bakelite where the creep is proportional to the load P. For the cases considered above where the creep is proportional to $(P - P_0)$, the general equation becomes

$$Y_t = \frac{F}{E}\left\{ P_t + \beta \int_0^\infty (P_{t-\omega} - P_0)\phi(\omega)d\omega \right\} \tag{9}$$

where only positive values of the integrand are, of course, to be taken.

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Relation of Warp Sizing to Loom Efficiency

By WILLIAM E. YELLAND *

A Research of U. S. Institute for Textile Research, Inc.

Summary

In a test covering a period of eight weeks the average loom efficiency of a group of looms was 90.4%. A careful analysis of the data collected during this test showed that the largest single cause of warp breaks was balling-up in back of the reed, which accounted for 23.1% of the warp stops or 10.1% of the total loom stops. If to this are added the number of other breaks occurring at the reed, i.e., those not due to balling-up, and the number of breaks due to frayed yarn, we have all the stops which can be considered as being caused by size failure. The results of this test showed that 37.59% of the warp stops, or 16.39% of the total loom stops, could be attributed in this way to size failure.

Moreover, supplementary tests showed that the time required to repair a warp break was approximately 2.19 times as long as that required for a filling break. So the failure of the warp size is an appreciable factor in the loss of loom efficiency even when the sizing is considered satisfactory by the weave room.

Next to size failure, knots were the most important source of warp stops. They accounted for 14.75% of the total loom stops.

Filling stops formed a large percentage: 56.37% of the total loom stops. Although this is an important cause of loss in weaving efficiency it is not as large as it might first appear to be, because of the fact that the average filling break takes less than one-half of the time required for repairing a warp break.

Conditions of Test

The test was made on 24 warps each 2400 yards long and containing 4296 ends of 150 denier 40 filament bright viscose rayon. The warps were prepared on the silk system and slashed with gelatin mixes on a 7-can rayon slasher under controlled conditions as described in a previous article of this series (Text. Rsch. Oct. 1940, *10*, P. 526–530).

They were woven in 24 cam looms with automatic bobbin change operating at 160 picks per minute. The fabric was a 3-leaf twill of construction 112 x 68. During the test period an operator followed the weaver

* Dr. Yelland was director at Massachusetts Institute of Technology of the filament viscose rayon phase on the warp sizing research from Jan. 1, 1936, to Sept. 30, 1938, and is now with Corn Products Refining Co., Research Laboratory, Edgewater, N. J.

around and recorded where possible the cause of each loom stop. The test extended through a period of eight weeks and covered 275 hours or 6600 loom hours.

A Comparison of Warp and Filling Stops

In the 6600 loom hours comprising the test there occurred 4717 loom stops (see Table 1), an average of 0.715 per loom hour. Of these 2058,

TABLE 1

Comparison of Warp and Filling Stops

Week of Test	1	2	3	4	5	6	7	8	Total
Test hours	23	40	40	40	34	34	32	32	275
Loom hours	552	960	960	960	816	816	768	768	6600
Warp stops	118	285	262	269	342	300	256	226	2058
Warp stops per loom hour	0.214	0.297	0.274	0.280	0.419	0.367	0.333	0.294	0.332
Filling stops	218	430	462	406	352	297	228	266	2659
Filling stops per loom hour	0.395	0.448	0.481	0.423	0.431	0.364	0.297	0.346	0.403
Total stops	336	715	724	675	694	597	484	492	4717
Total stops per loom hour	0.609	0.744	0.754	0.703	0.850	0.731	0.630	0.640	0.715
% Warp stops	35.1	39.9	36.2	39.9	49.3	50.3	52.9	45.9	43.69
% Filling stops	64.9	60.1	63.8	60.1	50.7	49.7	47.1	54.1	56.31
% Loom efficiency	91.20	90.25	92.72	90.81	87.66	88.68	90.75	91.31	90.42

or 43.63%, were warp stops, averaging 0.312 per loom hour. The filling stops were 2659, or 56.37%, an average of 0.403 per loom hour.

The warp stops varied between 0.2 and 0.3 per loom per hour except during the fifth, sixth and seventh weeks, when an epidemic of crossed ends occurred. These were traced to factors which had no relation to the size and hence could not be blamed on the size mixes.

Analysis of Warp Stops

An Analysis of Warp Stops is given in Table 2. They can be divided into six classes, as follows: (1) Balling-up at the reed; (2) yarn breaks at the reed for reasons unknown; (3) broken filaments; (4) knots from previous operations; (5) weaver's own knots; (6) miscellaneous.

(1) *Balling-up at the reed* is caused when one or more filaments are broken and stick out from the yarn. The low twist in the continuous filament yarn allows these filaments to be pushed back along the yarn, unless prevented by the gelatin or starch size, until a "fuzz ball" of appreciable magnitude is formed. These fuzz balls form chiefly in back of the reed. Occasionally they pass through the reed, but usually they remain in back of it and grow larger until they catch on the next yarn in the same dent in the reed. Then either of these yarns breaks and the loom stops. Fre-

TABLE 2

Analysis of Warp Stops. Per Cent of Total Warp Stops.

Group Week of Test	1	2	3	4	5	6	7	8	Average
(1) Balling-up at reed	19.49%	26.31%	24.81%	21.96%	22.22%	26.33%	14.06%	29.65%	23.10%
(2) At reed—reasons unknown	5.08	6.72	7.63	4.73	1.46	4.00	0.39	1.33	3.92
(3) Frayed yarn	12.71	8.07	6.87	9.80	14.03	10.66	16.01	6.19	10.57
Total	37.28	41.10	39.31	36.59	37.71	40.99	40.46	37.17	37.59
(4) Knots	29.61	11.23	19.08	19.26	15.20	14.66	15.23	19.03	17.91
(5) Weaver's knots	*	14.03	15.27	15.54	21.05	18.66	16.01	10.62	15.88
Total	29.61	25.26	34.35	34.80	36.25	33.32	31.24	29.65	33.79
(6) Miscellaneous	33.11	33.64	26.34	28.71	26.04	25.69	28.20	33.18	28.62

quently it is the yarn next to the one with the fuzz ball which breaks. Occasionally both yarns break.

Many of these fuzz balls were examined under the microscope and were found to be composed of one or more filaments matted together. There was no appreciable amount of size in them other than that already adhering to the filaments.

The formation of these fuzz balls is due to the inability of the size to protect the yarn against the chafing action of the loom and hence is a criterion for estimating the protective action of the size. It is possible to form a rough preliminary estimate in the following manner of the way in which the warp will weave: A sample yarn is taken from the warp and broken by hand in the usual way, whereupon the filaments will fly apart at the broken end. One of these filaments is held by the thumb and forefinger of one hand, or by means of forceps if more convenient, and an effort is made to separate the filament from the yarn by pulling the filament back along the yarn away from the broken end which is held by the thumb and forefinger of the other hand. *It has been observed repeatedly that, if pieces of filament two or three inches or more in length can be stripped off the yarn, the warp from which the sample was taken will give much trouble in the loom through balling-up at the reed. If, however, the size prevents pieces of filaments longer than an inch from being stripped off the yarn, the warp will probably weave well.* This test is crude but indicative of what may be expected of the warp in balling-up during weaving.

(2) A few yarns break at the reed for no apparent reason. That is, there has been no preliminary fraying evident to the eye, and there are no fuzz balls close by on which the break can be blamed. While these breaks cannot be attributed definitely to size failure they are grouped in this report with the breaks caused by balling-up at the reed. They form only a small percentage of the total number of warp breaks.

(3) A considerable number of yarns show fraying and break before they reach the reed. These breaks were classified under "broken filaments." It is a matter of dispute whether they are caused by size failure or are due to defective yarn.

(4) *Knots* were the cause of much loom stoppage. It was found desirable to divide them into two groups: those tied by the weaver in repairing warp breaks and those arising from previous operations. After the first week these two groups of breaks were itemized separately.

(5) *Weaver's knots* caused an appreciable percentage of warp breaks.

(6) *Miscellaneous* consists of all warp stops not classified in the five groups given above. It includes such diverse factors as the following: slubs, ends coming up, ends running out, rough shuttle, ends caught and crossed under other ends on the beam, broken heddle, slack end, waste in shed, and unknown. Although this class as a whole was the cause of approximately 30% of the warp stops no one factor was responsible for more than a very small percentage of warp stops. The relative importance of these varied from week to week. None of them could be traced to warp sizing.

Causes of Losses in Loom Efficiency

Examination of Table 2 shows that balling-up at the reed is the largest single factor responsible for warp breaks. Since the warp stops

are 43.63% of the total loom stops, balling-up is responsible for $0.231 \times 43.63\% = 10.1\%$ of the total loom stops.

Balling-up at the reed is the result of the chafing of the reed on the warp. The degree of balling-up is influenced partly by the mechanical quality of the original yarn, the kind of sizing, condition of drop wires and heddles on the loom, and the condition of the reed. Therefore, it is determined partly by the condition of the reed and partly by the warp size. It is doubtful whether the stops in groups (2) and (3) (see Table 2), can be blamed entirely on the size. Certainly none of those in groups (4), (5) and (6) can be attributed to the size. Therefore, in considering the effect of warp sizing on loom efficiency if the stops in groups (1), (2) and (3) are added together, we have all the stops that can possibly be attributed to the size in any way. From Table 2 it is seen that these three groups are resopnsible for 37.59% of the warp stops or $0.3759 \times 43.63\% = 16.39\%$ of the total loom stops.

TABLE 3

Cause of Stop	No. of Stops	% of Total Stops
Warp Size Failure	773	16.39
Knots	696	14.75
Miscellaneous	589	12.49
Filling	2659	56.37
Total	4717	100.00

When a break occurs at the reed, it necessitates the tying in of a length of sized yarn. This end of yarn, in many instances, must be threaded through the heddle and also through the reed. The weaver then holds the end and starts the loom and continues to hold on to the end for four or five picks. If a warp end breaks back of the harness, it requires a longer time to repair. The major number of filling breaks usually require simply a rethreading of the shuttle and the starting of the loom. In a supplementary test it was found that it took 2.19 times as long to repair a warp break as it did to repair a filling break. This is in accord with information kindly furnished us by Mr. Albert Palmer of Crompton and Knowles Loom Works. Mr. Palmer states that on constructions similar to that used in these experiments the time required to piece up a warp break is approximately two to three times that of a filling break.

Thus, although the number of stops due to size failure was 773 or 16.39% of the total loom stops their effect was equivalent to that of $773 \times 2.19 = 1693$ filling stops. In considering loss in loom efficiency the warp breaks must be weighted accordingly. An automatic loom is expected to operate at a loom efficiency above 90% at least; *i.e.*, a high standard of efficiency is expected. Therefore, when weaving on representative looms with fairly satisfactory warps, the effect upon loom efficiency of each individual defect is small. A loss in loom efficiency representing 16.39% of the total loom stops from failure of the size is of major importance, especially from the standpoint of cost.

One of the outstanding facts brought out by this survey is the importance of having a good running filling yarn. A loss of loom efficiency representing 56.37% of the total stops from filling trouble is a serious factor in mill costs.

Means of Evaluating Warp Sizing

It is evident that since warp breaks caused by balling-up at the reed are the only stops which can be attributed definitely and almost entirely to the size, they form a basis for evaluating sizes which is better than general loom efficiency alone.

In this series of tests all the warps were considered by the weave room to be satisfactorily sized, that is, the loss of weaving efficiency due to warp breaks was not greater than usual for this type of fabric. However, it was noted that in many cases the warp was damaged either during the warp break or during the operation required to repair the break. This damage frequently showed up as a defect when the greige goods were examined in the cloth room. It is evident, therefore, that a much more representative picture of sizing efficiency will be obtained if the figures secured in the weave room are supplemented by those on fabric grading and quality.

Dr. E. H. Killheffer Resigns Presidency of Institute

We announce with deep regret the resignation of our President, Dr. E. H. Killheffer, who wired that he was sick and tendered his resignation to the Board of Directors. The Board at a joint meeting with the Executive Committee in New York, January 10, 1941, formally accepted it and the following telegram was sent him:

> "Directors of U. S. Institute for Textile Research adopted resolution at meeting today, accepting with deepest regret your resignation as president, and expressing the hope that knowledge of your freedom from the duties of this office, which you have borne so long, will make for your speedy recovery."

Dr. Killheffer has served as president since May 24, 1938, and the Institute owes him a debt of gratitude for the generous personal contribution of time, effort and unflagging interest which he has made to the advancement and success of its work during the period of his office.

Dr. Louis A. Olney, First Vice President, by authority of the by-laws of the Institute will carry on the duties of the presidency until an election is held.

Microscopic Structure of the Cotton Fiber

By CHARLES W. HOCK, ROBERT C. RAMSAY, and MILTON HARRIS *

A Textile Foundation Publication

Abstract

During the examination of cotton fibers that had received various chemical treatments, a number of observations pertaining to the structural details of the fiber were made. Those phenomena which appeared to be new were investigated in detail. In addition, experiments described by earlier investigators were repeated in order to have a better composite picture of the structure of the fiber.

The cell wall of a cotton fiber consists of a primary and a secondary wall. The latter, which comprises the bulk of the fiber, consists of innumerable spirally oriented cellulose fibrils enclosed by a winding which also makes a spiral, but in the opposite direction from the former. Both the winding and the fibrils reverse their direction at frequent intervals along the axis of the fiber, their points of reversal being coincident. The secondary wall is enclosed by a thin primary wall. The latter is made up of fine crisscrossing strands of cellulose embedded in a membrane consisting principally of wax and pectic substance. The lumen also contains wax and pectic materials, plus various amounts of degenerated protoplasm.

When cotton fibers are swollen under certain conditions a lamellate structure is discernible in the secondary wall. The number of these lamellae increases with the age of the fiber.

On treatment of cotton fibers with cuprammonium hydroxide reagent, the cellulose dissolves, leaving residues which vary in amount and in structure, depending upon the extent of purification of the fibers. The residue from raw and from dewaxed fibers consists of fragments of the primary wall, and of a lesser amount of material from the lumen. The behavior of the fibers in the reagent depends in part on their maturity. Immature fibers containing only small amounts of cellulose swell relatively little in the reagent and the undissolved wax and pectic materials maintain the original tubular shape of the primary wall. When older fibers are given the same treatment they swell abruptly, thereby causing the primary wall to break in many places, giving rise to "balloons."

Irregular swelling along the fiber axis, which results in the formation of balloons, appears to be dependent in part on the orientation of the fibrils, and in part on the constricting influences of the winding and of the primary wall.

* Representing the Textile Foundation Research Associateship at the National Bureau of Standards.

I. Introduction

As part of a program of research relating to the chemical and physical structure of textile fibers, microscopic examinations were made of cotton fibers which had received various chemical treatments. A number of observations pertaining to the structural details of the cell wall of the cotton fiber were made. Those phenomena which appeared to be new were investigated in considerable detail. In addition, experiments described by earlier investigators (Balls,[1, 2, 3] Denham,[4] Sakostschikoff and Korsheniovsky,[5] Kerr,[6] Anderson and Kerr,[7] Farr,[8] Barrows[9]) were repeated, and are reported here for the sake of completeness.

II. Materials

Two varieties of *Gossypium hirsutum* L., Missdel–7 and Mexican Big Boll, were used in the investigation. Samples of each were furnished by the Agricultural Marketing Service of the U. S. Department of Agriculture. Missdel–7 was grown at the Delta Experiment Station, Stoneville, Miss. Only fibers from open bolls of this variety were used. The samples of Mexican Big Boll variety, grown at the Experiment Station at Raleigh, N. C., were from special case-historied material collected for joint fiber structural studies by the Bureau of Plant Industry and the Agricultural Marketing Service. The samples of the latter were principally young or immature fibers from bolls of known age. Fibers of different ages were obtained by tagging the flowers on the day of blooming and then collecting the bolls on successive dates so as to give material of the desired age. Two series of immature Mexican Big Boll fibers were used. One, collected during the summer of 1939, consisted of dried fibers in which deposition of secondary cellulose had begun. The other, collected during the summer of 1937, consisted of very young fibers with primary wall only. The second set of fibers was obtained from bolls, the locks of which, at the time of collection, had been placed in boiling water for a few minutes to untangle the fibers, and then stored in approximately 70 per cent ethyl alcohol.

Those fibers which received no chemical treatments are termed *raw fibers.* In the present paper the young Mexican Big Boll fibers which had been boiled in water and then placed in alcohol are also included in this category, although it is recognized that the treatment was not without effect on the composition of the fibers. The natural waxes were removed from some of the raw fibers by extraction with alcohol and ether for 24 hours each. These fibers are termed *dewaxed fibers.* A portion of the dewaxed material was further purified by extraction with alkali according to the method recommended for the preparation of standard cellulose. The procedure was essentially the same as that described by Corey and Gray[10] except that the apparatus of Worner and Mease[11] was employed. These fibers from which both wax and pectic substance were removed[12] are termed *depectinized fibers.*

Cuprammonium hydroxide solution was prepared according to the recommendation of Mease.[13] The solution contained 240 ± 5 g of ammonia (NH_3), 15 ± 0.1 g of copper, and 1.0 g of sucrose per liter. The concentration of nitrite was less than 0.5 percent. When not in actual use the cuprammonium hydroxide was kept under nitrogen in tightly stoppered bottles in the refrigerator.

Trimethylbenzylammonium hydroxide was used in some of the experiments. The stock solution, which was 2.5 N, was diluted with water just before using.

For staining cellulose, iodine and sulfuric acid,[14] zinc chloride-iodine,[14] and an alkaline solution of Congo red (0.5 percent of dye in a 0.5 percent solution of sodium hydroxide) were used. Aqueous solutions of ruthenium red were used for staining pectic compounds.[15] Although ruthenium red is not a specific stain for pectic substance, native pectic compounds invariably take a red color in the presence of this dye.[7]

III. Experimental Procedure and Results

The cell wall of the cotton fiber is composed of a primary and a secondary wall. The primary wall, in accordance with the description of Anderson and Kerr,[7] is considered to be the outer sheath of the fiber, or the portion of the wall formed as the fiber increases in length. Secondary wall then refers to that part of the wall which is laid down after the fibers cease to elongate appreciably. Only the thin primary wall encloses the protoplasm of the cotton hair as the fiber elongates during the first 13 to 20 days after its origin. Thereafter, the thickness of the wall is increased by a deposition of cellulose which comprises the secondary wall. For convenience in discussion, these two distinct layers are considered separately.

1. Primary wall

Young fibers, 3, 5, 8 and 15 days old, were mounted in water and examined microscopically. With ordinary light there was no evidence of structure in the thin primary wall. The walls stained deeply and uniformly with ruthenium red. When treated with an alkaline solution of Congo red, zinc chloride-iodine, or iodine and sulfuric acid, the fibers give a faint color reaction. In older primary walls, for example in a 15-day fiber, an ill-defined pattern was observable in the wall.

On examination with crossed nicols, unstained fibers showed practically no birefringence. Insertion of a selenite plate between the nicols produced faint colors indicative of a predominantly transverse orientation. Although these fibers showed little birefringence when unstained, they were clearly anisotropic after treatment with a dye such as Congo red. This increase in birefringence upon staining is due, apparently, to the double refraction of the preferentially oriented dye molecules.[16, 17]

When wax and pectic substance were removed from 15-day fibers the material which remained still had the outline of the original fiber, and stained with Congo red, zinc chloride-iodine, and iodine and sulfuric acid. Between crossed nicols the stained depectinized fibers were birefringent and showed a pattern like that in the stained walls of raw fibers. When the purified fibers were placed in cuprammonium hydroxide solution they appeared to dissolve completely in the reagent.

The stained walls of both the raw and the depectinized fibers were suitable for observing the cellulose * of the primary wall. In agreement with the description by Balls,[2, 3] and by Anderson and Kerr,[7] the cellulose, as observed with crossed nicols, was found to be present as criss-crossing

* While the identity of this material has not been definitely established, X-ray investigations, staining reactions, and behavior in cuprammonium hydroxide solutions indicate that it is probably cellulosic in nature.

strands. At 45° with respect to the planes of the nicols, the fiber showed its maximum brightness and presented a reticulate appearance (Fig. 1, A). When a fiber was rotated to the right or left of its position of maximum brightness, either one or the other of the systems of criss-crossing strands predominated the image (Fig. 1, B, C). Measurements * of the angles which these birefringent strands made with the axis of the fiber were made on fibers of different ages. The results indicated that the angle of orienta-

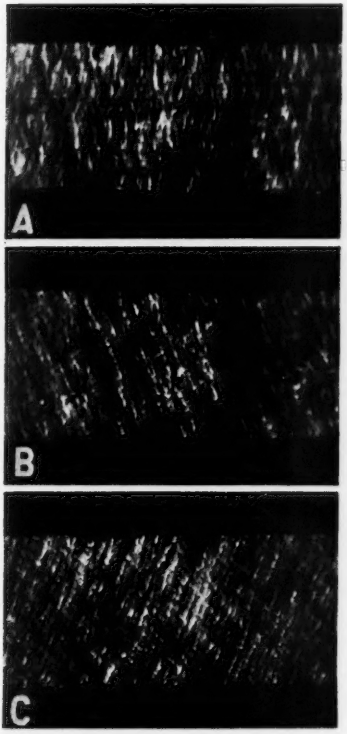

Fig. 1.

Raw 15-day cotton fiber stained with Congo red and photographed between crossed nicols.

A. Fiber at 45° with respect to planes of the nicols, showing criss-crossing strands of cellulose in the primary wall.

B. Same fiber placed at about 15° to the plane of light passing through the polarizer, showing one system of strands which predominate in this position.

C. Same as B, with fiber placed at about 15° to the plane of light passing through the analyzer, and showing the other set of cellular strands.

Magnification × 890.

tion decreased with increase in age of the fiber. In other words, the orientation was more nearly transverse in the younger than in the older ones. Likewise, the netlike structure of the cellulose, when the fibers were placed at 45°, appeared coarser (compare Fig. 1, A with Fig. 2, A), and showed greater birefringence in the older fibers. The greater birefringence of the older fibers is not readily apparent from the photographs as they were taken primarily to show the netlike structure. Measurements also indicated that

* The angle which the strands made with the axis of the fiber were measured at the center of the fiber by means of a rotating stage marked in degrees.

the angle which the cellulose strands made with the fiber axis was greater at the tip than at the base. These differences were especially noticeable in 15-day fibers where the growing cell had nearly attained its maximum length.

The above observations were confirmed by the examination of single walls obtained by cutting longitudinal sections of the fibers with the aid of a freezing microtome. Staining reactions and birefringence measurements gave essentially the same results as those obtained with intact fibers (Fig. 2, B).

After the initiation of secondary thickening, the predominantly transverse orientation (with respect to the fiber axis) of the cellulose in the primary wall was masked, after a few days, by the more nearly parallel orientation of the cellulose in the secondary wall. This change was evident upon examination of the fibers with crossed nicols and a selenite plate, as well as by observation with ordinary light, and is in agreement with the results of the X-ray work of Berkley.[18]

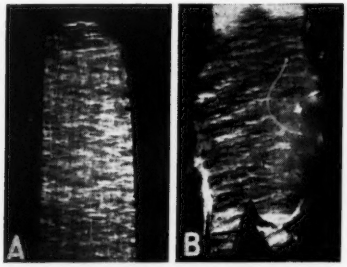

Fig. 2.

Raw fibers stained with Congo red and photographed between crossed nicols.
A. 3-day fiber at 45° with respect to the planes of the nicols, showing strands of cellulose in the primary wall.
B. Single wall of a 15-day fiber, showing strands of cellulose in the primary wall. Fiber placed at 45° angle with respect to the planes of the nicols. Magnification × 890.

2. Single fibers in cuprammonium reagent

The bulk of the cellulose of a cotton fiber is in the secondary wall. For this reason the attention of most investigators has been directed mainly to this part of the fiber.

In a previous investigation [19] it was found that the cellulose dissolved during the treatment of cotton with cuprammonium hydroxide solutions, leaving residues which varied in amount and in structure depending upon the extent of purification of the fibers. When *raw* and *dewaxed* mature fibers were placed on microscope slides and then treated with cuprammonium reagent, the fibers immediately began to swell and twist, often forming balloon-like structures (Fig. 3, A). The residue which remained after dissolution of the cellulose consisted principally of fragments (Fig. 3, B) of the primary wall and, to a lesser extent, of material from the lumen. These residues were isotropic, contained pectic substance,[12] and stained deeply with ruthenium red. They were often clumped into accordion-like structures which could be stretched with the needles of a micromanipulator

to form continuous tubes corresponding to the condition in the intact fibers. These tubes, which appeared to be soft and pliable, were readily torn by this treatment. *Depectinized* fibers dissolved in cuprammonium reagent, leaving only a negligible amount of isotropic residue which exhibited no definite cell structure.

When immature fibers in which only a small amount of secondary thick-

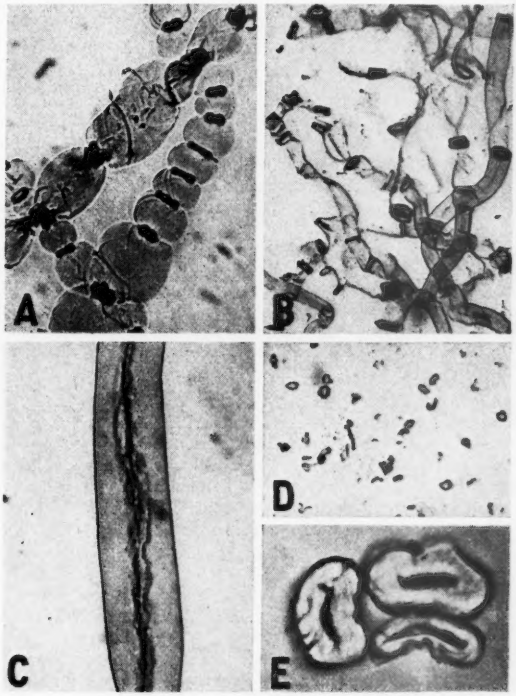

Fig. 3.

Raw cotton fibers in cuprammonium hydroxide solution.

A. Mature fibers swollen to form balloons.
Magnification × 120.

B. Residue which remains after the cellulose of raw mature fibers has dissolved in the reagent.
Magnification × 120.

C. Typical residue of a 22-day raw fiber after treatment with cuprammonium reagent.
Magnification × 500.

D. Residue which remains after treating cross sections of mature raw fibers with cuprammonium reagent.
Magnification ×120.

E. Raw cross sections of mature cotton fibers swollen slightly in dilute cuprammonium hydroxide solution, showing deeply stained material in the lumen and at the periphery of the fibers.
Magnification × 1220.

All the fibers were stained with ruthenium red.

ening had occurred were treated with cuprammonium reagent, relatively few visible changes took place. Few "ballons" were formed and therefore the primary wall was left practically intact. The mild reaction of young fibers in cuprammonium hydroxide was in sharp contrast to the swelling, writhing, and twisting of mature fibers. Young fibers containing only small amounts of cellulose swelled little in the reagent, and the undissolved wax and pectic materials maintained the original tubular shape (Fig. 3, C) of the primary wall. By noting their behavior in cuprammoium hydroxide, it was thus possible to distinguish mature from immature fibers.

3. Cross-sections of fibers in cuprammonium reagent

In order to study further the behavior of cotton fibers in cuprammonium reagent, cross sections of *raw, dewaxed,* and *depectinized* fibers were made. The usual procedure for making sections, whereby the fibers are embedded in paraffin, collodion, gelatin, or a similar material, could not be used as it is necessary to remove the embedding materials from the sections after they are cut, and the solvents used for this purpose also take out some of the waxes or pectic substance from the untreated cotton fiber. Accordingly, cross sections were made by simpler methods, which may not give sections as uniform as those obtained with embedded material, but have the advantage of leaving the fibers unaltered in their chemical composition.

By means of the device of Hardy,[20] fibers were cut into sections about 10μ thick without embedding. The sections were placed on microscope slides and then examined as cuprammonium hydroxide solution was drawn under the cover glass. The sections of *raw fibers* swelled in the reagent, and in a short time the cellulose appeared to be dissolved, leaving a residue in the form of rings and other fragments of material (Fig. 3, D) which stained with ruthenium red. Most of the residue came from the surface of the fiber, a lesser amount from the lumen. When this behavior was observed with crossed nicols, it was noted that the birefringence of the sections was lost as the cellulose dissolved, and that the residue was isotropic. When sections were swollen only slightly in dilute cuprammonium reagent and then placed in ruthenium red, deeply-stained red bands were discernible at the circumference and in the lumen (Fig. 3, E). Sections of *dewaxed fibers* behaved in essentially the same way as raw cotton. Dissolution of the cellulose took place rapidly, leaving an isotropic residue which stained with ruthenium red and was similar to that obtained from the raw fibers. When sections of *depectinized fibers* were treated with cuprammonium hydroxide they dissolved practically completely in the reagent.

Cuprammonium reagent dissolves the cellulose of single cotton fibers or of cross sections in a relatively short time. On the other hand, dilute solutions of the reagent swell the fibers but dissolution of the cellulose is greatly retarded. Swelling fibers in this way offers an excellent means of observing details of their structure. Cross sections about 10μ thick were placed on microscope slides, and then treated with cuprammonium hydroxide solution which had been diluted four to six times with concentrated ammonium hydroxide. The sections swelled to many times their original diameter, thereby revealing a lamellate structure (Fig. 4, A and B). During the course of swelling the sections usually inverted so that the region originally at the surface of the fiber occupied the former position of the lumen. Where inversion was incomplete, cone-shaped structures were formed (Fig. 4, C). Inversion was observed in sections of fibers from which the

wax and pectic substance had been removed, as well as in raw fibers. Very thin sections of mature fibers sometimes swelled without inverting. It should be pointed out, however, that a slight tear at the periphery, such as might easily occur in making sections only a few microns in thickness, would allow the sections to expand without inverting. Cross sections of

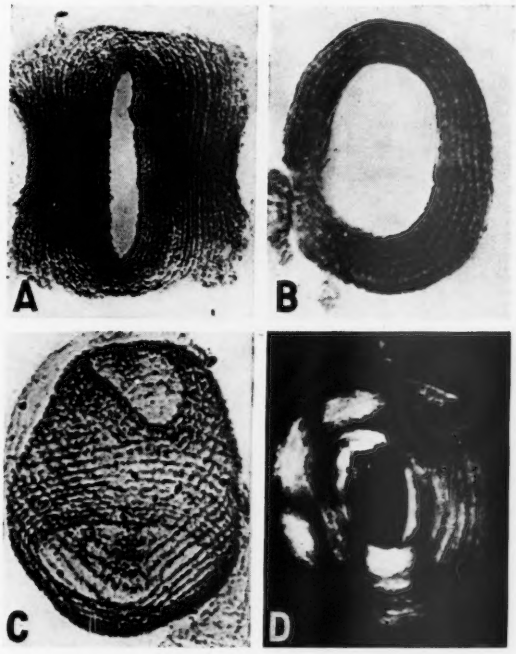

Fig. 4.

Cross sections of depectinized cotton fibers swollen in cuprammonium hydroxide solution to show lamellae.

A. Section of mature fiber showing lamellae.
Magnification × 420.

B. Section of immature (25-day) fiber showing lamellae.
Magnification × 945.

C. Section of mature fiber swollen to show lamellae, and partly inverted to produce a cone-shaped structure.
Magnification × 420.

D. Section of 26-day fiber showing lamellae. Photographed between crossed nicols.
Magnification × 700.

All the fibers were stained with Congo red.

immature fibers with only a small deposit of cellulose usually did not turn inside out. Apparently in immature fibers the lumen is sufficiently large to allow for expansion of the relatively small amount of cellulose. The regularity with which inversion accompanies the swelling of sections of mature fibers indicates that structural features of the fiber itself may be

responsible for this phenomenon. Since inversion occurs regularly, even in depectinized fibers, the insoluble wax and pectic substance in the primary wall cannot be primarily responsible for the observed behavior. It appears, therefore, that inversion may be due to a constricting influence, possibly of a layer or membrane which has not yet been identified, or what seems to be more probable to a differential rate of swelling between the inner and outer layers of the cellulose of the secondary wall.

4. Lamellae and growth rings

Lamellae were not readily seen in untreated cross sections but were clearly visible in properly swollen material. When treated with cuprammonium reagent diluted four to six times with concentrated ammonium hydroxide, swelling of the cellulose took place in such a way as to reveal lamellae. Slight pressure on the cover glass helped to spread the swollen sections and thus render the lamellae more distinct, but this technique was not necessary for making these structures visible. When, for example, the cover glass was supported by bits of broken cover glass to prevent pressure on the sections, swelling took place as usual and lamellae were clearly discernible. Likewise, when the sections were swollen in a hanging drop preparation * lamellae were similarly observed.

On treating the swollen sections with Congo red, alternating lamellae appeared to be deeply and lightly stained with the dye (Fig. 4, A and B). Between crossed nicols these sections showed alternating layers of strong and of weakly birefringent material (Fig. 4, D).

The above-described techniques were applied to Missdel-7 and to Mexican Big Boll cottons. In both varieties the structure of the fibers was essentially the same. The average number of lamellae in a mature fiber was between 45 and 50. Individual lamellae varied in thickness, from 0.1 to 0.2μ.† The first lamella appeared to be deposited between the eighteenth and twentieth day after flowering, the number increasing thereafter as the fiber approached maturity. The increase in the number of lamellae with age can be seen by comparing Fig. 4, A with Fig. 4, B.

The lamellae, so clearly observed in swollen cross sections, can also be seen in longitudinal view ‡ when the microscope is focused midway between the upper and lower surfaces of a swollen fiber. Fibers from which both wax and pectic substance were removed were preferable for these observations, as the purified fibers swelled more uniformly than raw fibers. When observed in this way the fibers showed striations ‡ running parallel to the fiber axis, and extending from the lumen to the outer surface (Fig. 5, A). The striations were readily discernible in unstained mounts, but the addition of Congo red rendered the pattern more distinct. When examined with crossed nicols alternating stripes of strong and of weakly birefringent material were observed (Fig. 5, B). As the fiber matures the thickness of the wall becomes greater and the number of striations is increased. The number of striations in Mexican Big Boll from 18 days to maturity was found to agree with the number of lamellae counted in cross sections.

* The sections were placed in a drop of dilute cuprammonium hydroxide solution on a cover glass, and the latter was inverted over a depression in a microscope slide.

† The width of the lamellae was obtained by dividing the thickness of the wall of the unswollen fiber by the number of layers counted after swelling.

‡ In the present paper, the layers observed in cross section are called lamellae, whereas the layers observed in longitudinal view are called striations. Both lamellae and striations refer, however, to the same structures.

According to Kerr,[6] two lamellae, one compact and one porous, are deposited each day during the period of secondary wall deposition. These two lamellae (or two adjacent striations) together constitute a daily growth ring. Using "growth ring" in this sense, the relationship between these rings and the age of the fiber is shown graphically in Fig. 6. It is readily seen that there is a close correlation between the counts of lamellae and of striations. For the first 10 to 15 days after the initiation of secondary deposition, one growth ring was laid down per day. Thereafter, until maturity, additional growth rings were formed but the rate of one ring per day was apparently not maintained. However, closer examination of the

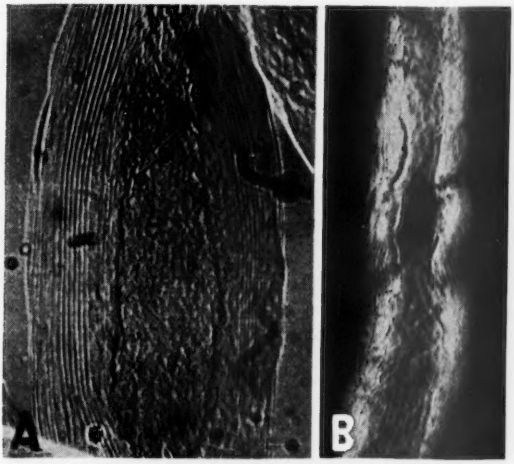

Fig. 5.

Depectinized cotton fibers swollen in cuprammonium hydroxide solution to show striations.
A. 51-day fiber showing striations.
Magnification × 500.
B. Mature fiber showing striations. Photographed between crossed nicols.
Magnification × 400.
All the fibers were stained with Congo red.

data showed that the counts of lamellae and of striations varied considerably in fibers older than 35 days. This may mean that individual fibers continue to deposit one growth ring daily but that the average for the group is lowered because of the early maturation of some of the fibers. These results confirm the work of Balls[1] and Kerr;[6] they are not in agreement with the results of the investigations by Sakostchikoff and Korsheniovsky[5] and Barrows[9] who were unable to establish a consistent relationship between the number of lamellae and the age of the fibers.

Fig. 7 shows the increase in width of the secondary wall with the age of the cotton fiber. After about the first 15 days of secondary wall formation there is only a slight increase in thickness as the fibers continue to mature. This is consistent with a recent investigation[21] in which it was demonstrated that the percentage of cellulose in a series of cotton samples of

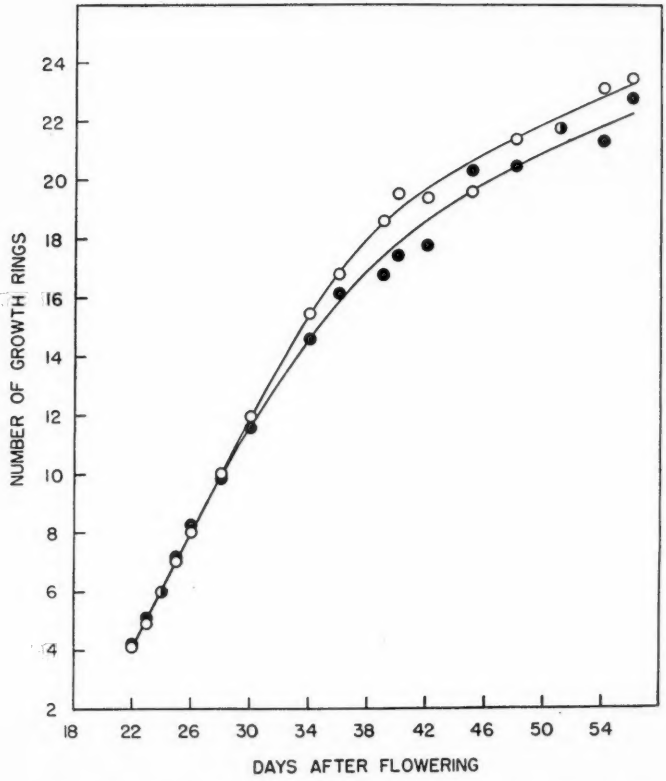

Fig. 6.

Growth rings in relation to the age of the fiber. The open circles represent growth rings estimated from the number of lamellae counted in fiber cross sections (25 sections counted for each point of the curve). Solid circles represent growth rings estimated from the number of striations counted in longitudinal view (50 sections counted for each point of the curve).

different ages increased rapidly until about the 35th day after flowering, and that thereafter there was only a small further increase with age. A comparison of the curves of Figs. 6 and 7 suggests that the initial growth rings are wider than those laid down after the 35th day.

Lamellae, or striations, were also revealed when fibers were treated with reagents other than cuprammonium hydroxide. Trimethylbenzylammonium hydroxide had essentially the same effect as cuprammonium reagent. Combined treatment with sodium hydroxide and carbon disulfide, and treatment with phosphoric acid also revealed a lamellate structure in the fiber, but cuprammonium hydroxide and trimethylbenzylammonium hydroxide were preferable for this work.

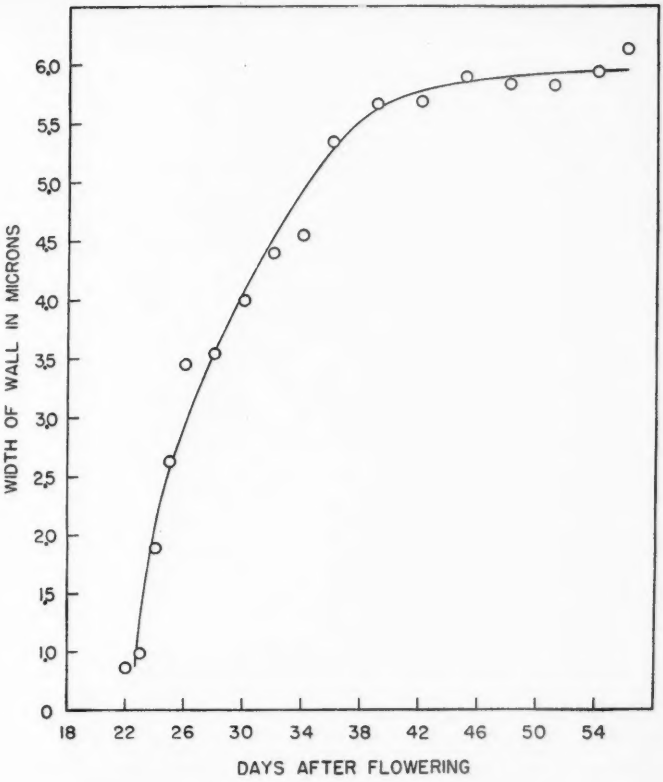

Fig. 7.
Increase in width of the wall with increase in age of the fiber.
Each point of the curve represents an average of 200 measurements.

5. Fibrillar structure

The investigation of the structural details of the cotton fiber was continued with single fibers. These were mounted in distilled water (Fig. 8, A), and then examined with crossed nicols. When the fibers were placed approximately parallel to the plane of vibration of light through one of the prisms, the high birefringence of the fiber was interrupted by dark extinction bands at irregular intervals along its axis (Fig. 8, B). The same phenomenon has previously been noted by Balls.[2] When a selenite plate was inserted between the nicols, contrasting colors, indicative of differences in orientation, appeared in regions adjacent to the extinction bands (Fig. 8, C). The number of convolutions or twists of the fiber which occurred in a region between two bands of extinction varied considerably, depending apparently

on the length of the region. However, in a region which showed uniform optical properties the convolutions were always in the same direction.

The optical variations just described were also correlated with differences in structure as observed in depectinized * fibers swollen in dilute cuprammonium hydroxide or in solutions of trimethylbenzylammonium hy-

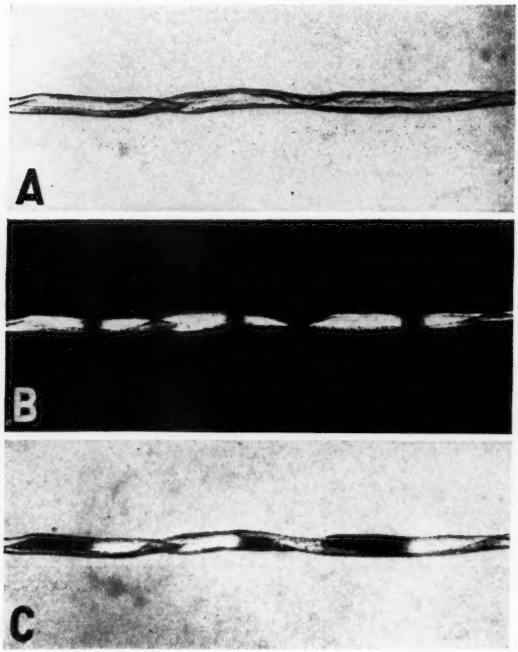

Fig. 8.

Raw mature fiber mounted in water and photographed under various lighting conditions.

A. Ordinary light, showing convolutions in the fiber.

B. Between crossed nicols, showing bands of extinction where the fibrillar orientation reverses.

C. Between crossed nicols and with selenite plate, showing the effect due to differences in color in regions adjacent to the extinction bands.

Magnification × 180.

droxide. For this experiment the cuprammonium reagent was diluted 3 to 4 times with concentrated ammonia and the trimethylbenzylammonium hydroxide was diluted with water to a concentration of 2.2 to 2.4 *N*. Upon swelling, the fibers clearly revealed a fibrillar structure. The bulk of a fiber was made up of innumerable fine fibrils oriented at an acute angle with

* Depectinized fibers were best suited for these experiments, since, after removal of the outer membrane of wax and pectic substance, irregular swelling along the axis could be minimized.

respect to the axis of the fiber. The fibrils were enclosed by a winding * which appeared to be a little coarser than the fibrils themselves.

In fibers which had been swollen only slightly, it was observed that this outer winding was closely spaced and made a moderately steep spiral (Fig. 9, A and B). The winding, like the bulk of the fiber, stained with Congo red, zinc chloride-iodine, and iodine and sulfuric acid, and appeared to dissolve in cuprammonium reagent. It made either an "S" or a "Z"

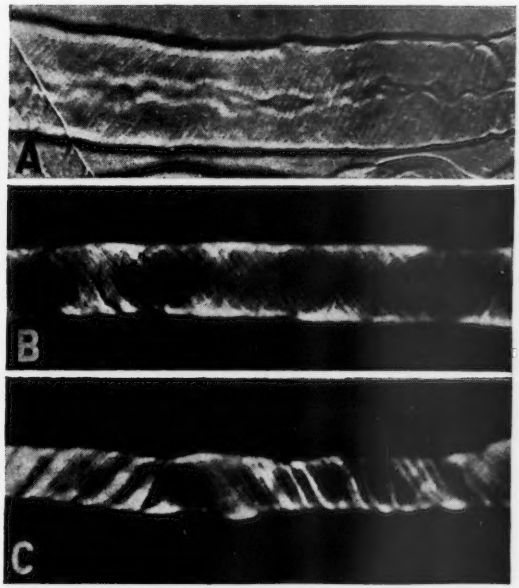

Fig. 9.

Depectinized fibers showing the winding.

A. Fiber swollen slightly in dilute cuprammonium hydroxide solution.

B. Fiber treated with zinc chloride-iodine and photographed between crossed nicols.

C. Fiber showing reversal, treated with zinc chloride-iodine and photographed between crossed nicols.

Magnification × 420.

twist † around the fiber, reversal of direction taking place (Fig. 9, C) many times in a single fiber. Examination showed that both types of twist occur with about equal frequency. Invariably the orientation of the fibrils immediately beneath the outer winding was in the opposite direction. In other words, if the winding made an "S" twist, the fibrils immediately beneath made a "Z" twist. As far as could be determined, all of the fibrils under the winding at any one place appeared to run in the same direction.

* Although the winding closely resembles the fibrils which make up the bulk of the fiber, to facilitate discussion it will be considered separately.

† According to A. S. T. M. Standards on Textile Materials [22] a yarn or cord has an S twist if, when held in a vertical position, the spiral conforms in slope to the central portion of the letter "S," and a Z twist if the spirals conform to the central portion of the letter "Z." In the present paper the same terminology will be applied to fibrils.

It became apparent that reversals of direction of the cellulose fibrils and of the winding were responsible for the optical differences observed with crossed nicols, and that the band of extinction was the place at which the reversals occurred. The number of optical reversals occurring in a single fiber or fragment invariably was the same as the number of fibril

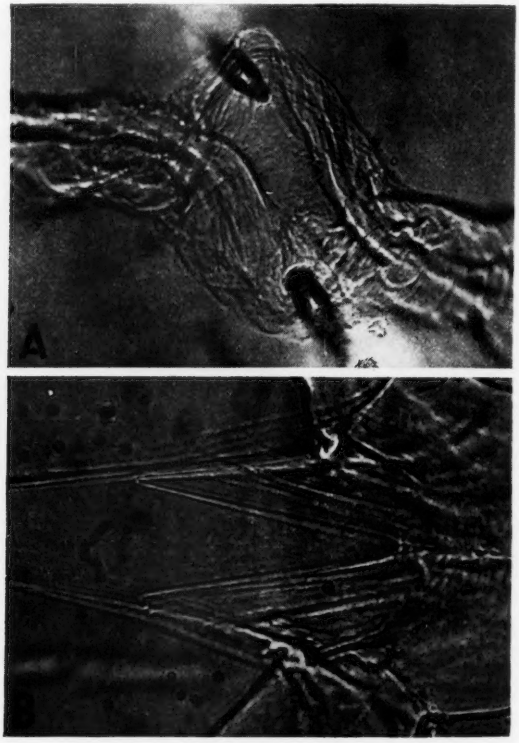

Fig. 10.

Depectinized fibers swollen in dilute cuprammonium hydroxide solution and handled with micro-needles.

A. A single fiber being dissected with micro-needles to show fibrillar structure.

B. Fibrils being pulled away from the fiber. (The needles are not shown in the photomicrograph.)

Magnification × 500.

reversals. Likewise, the relationship between the fibrillar orientation and the convolutions became clear. It was found that the direction of a convolution usually was the same as the spiral direction of the winding. That is to say, when the winding made an "S" twist around the axis, the convolutions in that region also showed an "S" twist.

The fibrillar structure of the secondary wall was studied to better advantage upon handling the fibers with mieroneedles. In some cases the outer winding was picked up by the needles and pulled away from the rest of the fiber, or the winding was pulled along the axis of the fiber in such a way as to form a clump. Needles were brought into the innumerable fine fibrils comprising the bulk of the cellulose of the secondary wall only by careful manipulation. The difficulty in picking up these fibrils appeared to be due to lateral cohesion of the fibrils themselves. After insertion of the needles, however, the fibrils could be separated from one another (Fig. 10, A and B) and otherwise subjected to mieromanipulative methods.

6. Balloon formation

Depectinized fibers were also examined in cuprammonium hydroxide solutions of various concentrations. It soon became clear that there was a relationship between the optical differences and fibril reversals on the one hand, and balloon-formation on the other. In *concentrated* cuprammonium hydroxide solutions (reagent diluted not more than 3 times with concentrated ammonia) dissolution of the cellulose of the fibers took place in a relatively short time. The fibers appeared to break into large chunks which became progressively smaller as dissolution proceeded. Observed between crossed nicols the highly birefringent fibers were seen to break up into fragments which steadily became less birefringent until the field of the microscope was black. In *dilute* solutions (diluted between 3 and 10 times) the fibers swelled greatly but complete dissolution generally did not occur. As swelling took place balloons were occasionally formed. When the behavior of the fibers was observed with crossed nicols the fibers were seen to become progressively dimmer as the cellulose dissolved. The expanded part of a balloon was the first to lose its birefringence, since here the cellulose had become highly disorganized. On the other hand, the constrictions were slow in losing their anisotropy (Fig. 11, A), since the reagent penetrated these regions slowly. When fibers which had lost much of their birefringence during swelling in cuprammonium solution were placed in water, some of the original birefringence of the fibers, as well as some of their fibrillar appearance, was restored.

The occurrence of balloons in *depectinized* fibers was somewhat surprising, since experiments by ourselves and by others indicated that the primary wall plays an important role in the formation of balloons. Accordingly, the phenomenon was studied further, especially in depectinized fibers from which the wax and pectic substance had been removed. The optimum concentration of cuprammonium hydroxide solution for observing balloons in these fibers was between 15 and 20 percent (1.5 to 2.0 ml of the reagent diluted to 10 ml by the addition of concentrated ammonia). When depectinized fibers were placed in these solutions they often swelled unevenly along the axis, thereby giving rise to balloons (Fig. 11, B and C). The expanded portion of the balloons almost invariably occurred between the points at which the fibrils reversed, whereas the reversal points themselves formed the constrictions between adjacent balloons. Fragments of depectinized fibers which showed no extinction bands usually swelled without the formation of balloons, and the direction of the fibrils was found to be constant. Because of the orientation of the cellulose within the fiber, the greatest swelling occurs perpendicular to the fiber axis, while at the same time there is an actual decrease in fiber length. As these changes in length

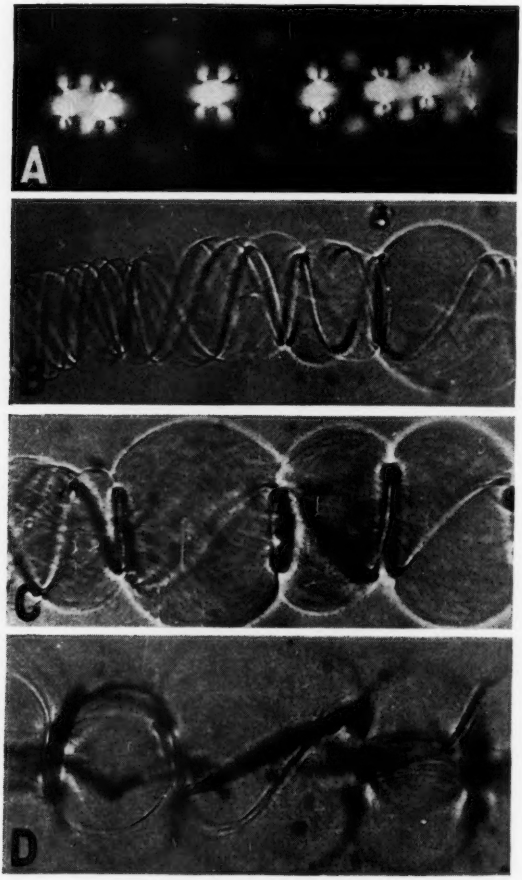

Fig. 11.

Cotton fibers swollen in cuprammonium hydroxide solution to produce balloons.

A. Depectinized fibers showing balloons, photographed between crossed nicols. (Selenite plate inserted.)
Magnification × 260.

B. Depectinized fibers showing winding. The coarse appearance of the winding may have resulted from the coalescence of individual fibrils during incipient balloon-formation.
Magnification × 420.

C. Typical string of balloons in a depectinized fiber.
Magnification × 500.

D. String of balloons in a raw fiber showing the winding and fragments of the primary wall connecting the constrictions. Fiber stained with ruthenium red.
Magnification × 440.

and in width take place, the winding assumes a more transverse position (Fig. 11, B) and becomes clumped at the constrictions. However, the uniform lateral expansion of the fiber is hindered somewhat by this same winding which does not expand freely in that direction. Accordingly, the winding also appears to exert a constricting influence on the swelling of the fibers.

It has been recognized that the primary wall is responsible, at least in part, for the formation of balloons. The rupture of this wall into a series of constrictions which restrains the expanding cellulose, has frequently been described. Furthermore, fragments of *raw* fibers which show uniform optical characteristics often produce many balloons as swelling occurs, whereas similar fragments of depectinized fibers do not produce balloons. When balloons are formed in raw fibers the cellulose fibrils may push through the winding and the primary wall, so that a constriction consists of a clump of the outer winding as well as of a portion of the primary wall (Fig. 11, D). Thus, the above experiments suggest that the irregular swelling along the fiber axis, which frequently results in balloon formation, is dependent in part on the orientation of the fibrils and in part on the constricting influences of the winding and of the primary wall.

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Abstracts

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I. Fibers (Synthetic and Natural) and Fiber Analysis

Constitution of Animal Textile Fibers. A. Wahl. *Rev. gén. mat. color*, V. 44, P. 41–5 (1940); abs. in *C. A.*, V. 34, Col. 6081.

A review of the results obtained by chem. and x-ray methods, with special reference to silk fibroin. X-ray photographs of ramie, fibroin, wool (normal and stretched), and asbestos fibers are given. (C)

Denaturation of Sericin. I. Denaturation of Sericin Caused by Boiling in Hot Water. Zirō Hirose. *J. Agr. Chem. Soc. Japan*, V. 16, P. 209–12; *Bull. Agr. Chem. Soc. Japan*, V. 16, P. 43–5 (in English) (1940); abs. in *C. A.*, V. 34, Col. 5666.

Sericin in the outside cocoon layer adsorbed more Cr_2O_3 by treating with basic Cr sulfate than that in the inside layer. When the cocoon layer was boiled with H_2O for 15–30 min., sericin retained in the cocoon absorbed more anionic chromate complex (basic oxalatochromate) and less cationic chromate complex (basic Cr sulfate) than the original one. (C)

The "Feltability" of Cellulose as Explained by the Micellar Structure of the Fibers. G. Centola. *Atti X° congr. intern. chim.*, V. 4, P. 138–44 (1939) (in Italian); abs. in *C. A.*, V. 34, Col. 5277.

C. studied the tensile strength of fibers treated with NaOH and KOH of various concns. He believes that in the process of refining macromols. become partly detached from the main fibers and thus form a system of tentacles surrounding the fibers. In the felting process sheet formation is due to the stabilization of the attractive forces between the hydroxyl groups of those macromols. (C)

II. Yarns and Fabrics

Fiber Injury Due to Mechanical Influences during Laundering. Klara Schönleber. *Mell. Textilber.*, V. 21, P. 228–33 (1940); abs. in *C. A.*, V. 34, Col. 6087.

Fabrics of linen (I), staple rayon (II) and rayon 16%-cotton 84% (III) were machine laundered under the following conditions: (1) loose, (2) enclosed in a net, (3) with the machine running continuously, (4) with the machine running only 19 min. per hr. total washing time. After 50 washings the goods were examd. for loss of strength and for injury to the individual fibers by means of photomicrographs. On II continuous running caused much greater damage than intermittent running, on I less and on III least comparative damage. On III enclosure in a net caused more injury to the fibers than continuous running of the machine. (C)

Impregnation of Linen Fabrics for the Prevention of Rotting. A. P. Rufimskiĭ. *L'no-Pen'ko-Dzhutovaya Prom.*, 1938, V. 8, No. 5, P. 46–7; *Chem. Zentr.*, 1938, II, 3629; abs. in *C. A.*, 1940, V. 34, Col. 6453.

Expts. on the impregnation of linen fabrics with $CuSo_4 \cdot 5H_2O$ and $K_2Cr_2O_7$ showed the amt. of Cu absorbed to be greater than the amt. of Cr. No Cu was found in the spent bath although 1.34 g. $K_2Cr_2O_7$ per l. was still present. The salts taken up by the fibers were washed out again by subsequent washings. On the finished fabric 0.35% $CuSO_4 \cdot 5H_2O$ and 0.03% $K_2Cr_2O_7$ with a moisture content of 5.49% could be detected. The most satisfactory salt concn. in the bath is given as 5 g. Cu salt and 2.5 g. Cr salt per l. (C)

Isoelectric Washing of Wool. E. Elöd. *Mell. Textilber.*, V. 20, P. 569–70 (1939); cf. *C. A.*, V. 33, Col. 9001; *Ibid.*, V. 34, Col. 5285 (1940).

Evidence showing that the isoelec. point of wool is at pH 4.9 is reviewed. Tendencies toward degradation of wool during processing are reduced to a min. by washing, dyeing or otherwise treating and finishing wool at a pH as near 4.9 as is practical. This is due to the fact that at wool's isoelec. point its swelling is reduced to a min. and hence susceptibility to mech. or chem. attack is also at a min. (C)

Observations on the Tendering of Silk. L. DaCosta Ward. *Can. Text. J.*, V. 57, No. 14, 28, 30, 36 (1940); abs. in *C. A.*, V. 34, Col. 6084.

Investigations of tests for pH and NaCl, viscosity of silk after exposure, strength tests on silk, and the effect of dyes and sunlight show that the tendering of silk fabric is due to the destructive action of sunlight accelerated by accumulated chlorides acquired principally from "salt air." (C)

Rayon Plant Capacity, and Outlook. Stanley B. Hunt. *Text. Wld.*, Sept. 1940, V. 90, P. 90–1.

The present situation and some factors bearing on future plant expansion. (C)

Staple Fibre Yarns: Direct Production. A. Jaumann. *Kunstseide*, 1940, V. 22, P. 66–70, 98–104; abs. in *J. T. I.*, July 1940, V. 31, P. A364.

The direct use in the weaving of carpets of ropes of continuous rayon filaments obtained by twisting together the filaments from several spinning

nozzles is discussed, and various shortened processes for the production of staple fibre yarns from continuous filaments are briefly described. The methods include the direct production of staple fibre yarns on the spinning machine by staggered cutting of the filaments on the way to the spinning pot, the breaking of surface filaments of continuous filament yarns by passing over rough or sharp edges, the production of yarns on machines comprising a combination of a card with web divider rubber leathers and a spinning device, the production of yarn with the aid of web dividers, shortened processes for the production of slivers, and processes depending on the feeding of the continuous filaments to drawing or speed frames provided with cutting devices. (C)

"Sylbroc" Yarns and Fabrics. *Text Mfr.*, July 1940, V. 66, P. 268.

A new yarn of spun silk and cut rayon has approximately the full qualities of real spun silk with considerable economies. (C)

Synthetic Fibers Enlisted. *Text. Wld.*, Sept. 1940, V. 90, P. 86–89.

A series of articles demonstrating how synthetic fibers will assure an adequate supply of textiles for both military and civilian purposes. (C)

Treatment of Cellulose Fibre Materials. "Celloganist." *Silk J. & Rayon Wld.*, June 1940, V. 17, P. 19–20.

A new line of development in the treatment of cellulose fibres is opened up by the recent discovery of commercial methods to manufacture special organic bases comparable to the older inorganic bases such as caustic soda and potash. This article describes these new bases and indicates their use in mercerizing cotton, the manufacture of viscose rayon, and in cellulose viscosity measurements. (C)

"Tropal" Interlining Fabric. Airpak Ltd. *Text. Mfr.*, 1940, V. 66, P. 179; abs. in *J. T. I.*, July 1940, V. 31, P. A376.

"Tropal" fabric consists of a carded web of "tropical fibre as light and lustrous as kapok" between two layers of very open muslin, the whole being stitched together in lines about ¾ inch apart. It is usually made 39 inches wide and in rolls 125 yds. long. The fine fibres are said to be hollow, water repellent and rot resisting. The material is included in Air Force flying kit, being light and warm, and buoyant in water. (C)

Water Repellency of Cotton Hose. M. S. Furry and L. E. Weidenhammer. *Rayon Text. Mo.*, June 1940, V. 21, P. 72–3; July 1940, V. 21, P. 73–4. (C)

Water-Resistant Finishes (for Textiles). C. A. Norris. *Text. Mercury and Argus*, Jubilee No., Sept. 1939, P. 173–7; abs. in *C. A.*, V. 34, Col. 4582.

The development of H_2O-repellent finishes from the two-bath Al + soap method to those using wax + Al acetate emulsions (Ramisit and Cerol) and quaternary NH_4 compds. (Velan) is reviewed. (C)

III. Chemical and Other Processing (Not Otherwise Classified)

Low Wear Resistance of Dyeings in Acetate Rayon and Acetate Rayon Mixtures. Carl Blau, Jr. *Fäber- u. Chemischreiniger*, 1939, P. 129–30; *Chem. Zentr.*, 1940, I, P. 645; abs. in *C. A.*, 1940, V. 34, Col. 4914.

Causes of defects are excessively concd. dyeing baths, presence of Ca in water, incorrect dyeing methods and excessive amts. of dyes. Faulty dyeings can be corrected by aftertreatments with soap baths contg. Na phosphate. (C)

Naphtols AS: Analysis. K. D. Chtcherbatchev. *Prom. Organ. Khimii*, 1939, V. 6, P. 249–352 (through *Chim. et Ind.*, 1940, V. 43, P. 670); abs. in *J. T. I.*, July 1940, V. 31, P. A385.

Naphtols AS couple quantitatively with diazobenzene in pyridine solution. They may be determined in the presence of β-hydroxynaphthoic acid by precipitation by means of carbon dioxide in the presence of caustic soda, filtering, dissolving the precipitate in pyridine and titrating the arylide with a solution of diazobenzene. Another sample of the original Naphtol AS is dissolved in pyridine and titrated directly with diazobenzene. The difference between the two titrations can then be used to calculate the percentage of β-hydroxynaphthoic acid. (C)

Organic Substances that Cause Swelling. J. R. Katz and J. Seiberlich. *Rayon Text. Mo.*, January 1940, V. 21, P. 82–3. (C)

Processing, Dyeing and Finishing Lanital Mixed with Wool. Edgar Riedel. *Mell. Textilber.*, V. 20, P. 507–11 (1939); abs. in *C. A.*, V. 34, Col. 3921 (1940).

This is a discussion of precautions to be observed when textiles contg. lanital (I) are sized, carbonized, dyed or otherwise processed because of its low wet strength and its great sensitivity to alkalies. Methods of manufg. I are also discussed with photomicrographs showing the differences in phys. structure between I and wool. (C)

Qualitative Identification and Quantitative Estimation of Nylon in the Presence of Silk, Wool and Cotton. R. Gerber and K. Lathrop. *Am. Dye. Rptr.*, Sept. 2, 1940, V. 30, P. 437–440, 457.

Summary. 1. In undyed fibre mixtures nylon is most readily distinguished by dyeing with neocarmine. 2. Chemically nylon is best distinguishable from (a) Cotton, by the solubility of nylon in hot concentrated acetic acid. (b) Silk, by the insolubility of nylon in calcium thiocyanate, sp. gr. 1.20–1.21 at 70° C. (c) Wool, by the insolubility of nylon in boiling 5 per cent potassium hydroxide. (d) Cellulose acetate, by the insolubility of nylon in acetone. (e) Regenerated celluloses, by the insolubility of nylon in calcium thiocyanate of sp. gr. 1.36 at 70° C. 3. The use of calcium thiocyanate, sp. gr. 1.20–1.21 at 70° C., to remove silk in the analysis of a silk-nylon mixture is satisfactory. 4. The use of boiling 5 per cent potassium hydroxide to remove wool in the analysis of a wool-nylon mixture gives accurate results. 5. The analysis for nylon in cotton-nylon mixtures by the solution of nylon in boiling 80 per cent acetic acid gives satisfactory results. (C)

IV. Research Methods and Apparatus

Microscopic Investigation of Pure Wool Cloth. Chemical and Microscopic Determination of the Addition of Casein Wool. J. Straub and G. J. van Amerongen. *Chem. Weekblad,* V. 37, P. 236–8 (1940); abs. in *C. A.,* V. 34, Col. 6083.

A method is described for assaying the quality of the wool in military cloth by microscopic examn. The method was applied to cloths into which 30% of casein wool had been woven and gave correct results for the casein wool content. The microscopic results were checked by S detns. Photographs are given which show the difference in structure between natural-wool and casein-wool fibers. (C)

Staple Fibre: Determination in Mixtures with Vegetable Fibres. A. Cappelli and R. Tuffi. *Boll. Assoc. Italiana Chim. Tess. e Color.,* 1939, V. 15, P. 111–117; abs. in *J. T. I.,* July 1940, V. 31, P. A395.

Details are given of a method for the determination of staple fibre in yarns and fabrics in which it is mixed with cotton or other vegetable fibres. The sample is first treated with dilute sodium carbonate solution and dilute hydrochloric acid and then for 1½ min. in a boiling solution of basic zinc chloride having a specific gravity of 1.835 and a boiling point in the neighborhood of 135° C. This treatment dissolves the staple fibre. Data showing the losses in weight suffered by vegetable fibres in such treatment are given; the loss shown by cotton is low enough to be neglected in the calculations. Results of determinations on staple fibre-cotton fabrics of known composition are given. The method is claimed to be rapid and accurate. (C)

Synthetic Resin Finishes: Detection. H. Rath. *Textilber.,* 1940, V. 21, P. 175–76; abs. in *J. T. I.,* July 1940, V. 31, P. A399; *C. A.,* V. 34, Col. 5667.

Methods for the detection of modern finishes of the resin type on textile materials are briefly described. Finishes with a urea- or thiourea-formaldehyde basis evolve formaldehyde on boiling with either acids or alkalis and the formaldehyde can be detected by the carbazole reaction. Finishes with an aldehyde basis (excluding urea or thiourea) on cellulose materials evolve formaldehyde on boiling with acids but not on boiling with alkalis. Finishes comprising polymerization products of the ethylene series (polyacrylic acid ester, polyvinyl acetate, etc.) can be detected by an intense yellow coloration of the materials on dyeing with Celliton fast yellow RR, the dyeing being fast to treatment with warm soap solution. (C)

Technical Evaluation of Textile Finishing Treatments. III. Use of Rank Correlation for Comparison of Data. Lelia J. Winn and Edward R. Schwarz. *Text. Rsch.,* Sept. 1940, V. 10, P. 453–461; *Am. Dye. Rptr.,* Aug. 5, 1940, V. 29, P. P400–P404.

The nature and use of the Kendall coefficients of rank correlation and of concordance are presented with details of the methods of computation involved. They are found to be superior to the Spearman coefficient previously used both as a matter of convenience and of statistics. This discussion is given in anticipation of the papers immediately following in this series which will make frequent use of the Kendall rating system. (C)

TEXTILE OILS: ALKALINE AGEING TEST. W. Garner. *Analyst*, 1940, V. 65, P. 219–220; abs. in *J. T. I.*, June 1940, V. 31, P. A337.

The test is designed to give visual evidence of the formation of undesirable end-products by oils unsuitable for lubricating textile fibres. Small samples (3 inches square) of white worsted serge are spotted with oil, aged (4 hours at 80° C.) in an atmosphere of ammonia, scoured and dyed with Coomassie Blue R. L. S. The discoloration produced by alkaline ageing is due (a) to oxy-acids (brownish salts) and (b) to yellowish condensation products; the former are scoured out easily, but the latter form resists. An oil which produces a resist greater than that given by a good olive oil should be considered unsuitable for textile use. (C)

A TIME-TEMPERATURE CONTROL FOR SULPHITE DIGESTERS. John F. Inderdohnen. *Paper Trade J.*, 1940, V. 111, P. 31–4.

In this paper a new recording and controlling potentiometer instrument is considered and its mechanical and operating principles explained. The instrument eliminates the complicated moving mechanisms usually employed in instruments of this type. It is accurate and sensitive and its ruggedness renders it adaptable to applications in the pulp and paper industry where potentiometer type instruments have heretofore not been considered suitable. (C)

USE OF THE POLARIZATION MICROSCOPE IN THE INVESTIGATION OF FIBRES. A. Herzog. *Melliand Textilber.*, 1940, V. 21, P. 97–105; *J. Soc. Dyers Col.*, June 1940, P. 288; abs. in *C. A.*, 1940, V. 34, Col. 4578.

A detailed and comprehensive survey is given of the applications of the polarization microscope in the analysis of textile fibres, e.g., in the detection of natural silk and rayon, the differentiation between mercerized and unmercerized cotton, in determining the degree of nitration of cotton, in examining the shape of fibre cross-sections, and in the microchemical examination of fillers, impregnating material, etc., in paper and textiles. Excellent photo-micrographs are included. (C)

WATER ABSORPTION OF LOOSE STAPLE RAYON: TESTING DEVICE FOR DETERMINATION OF. H. Bundesmann. *Deut. Wollen-Gewerbe*, 1939, V. 71, P. 1208–10; abs. in *C. A.*, 1940, V. 34, Col. 1493.

An illustrated discussion. (C)

WOOL: DETERMINATION OF DAMAGE IN. Gross, Roll, and Schreiber. *Textilber.*, 1939, 20, 357. *Text. Col.*, August 1939, V. 61, P. 545–6.

A. J. Hall summarizes the test, which is based upon the fact that damaged wool is more reactive towards a diazotized amine than it is to a solution of diazotized sulphanilic acid which is the basis of the Pauly test. (C)

WOOL: PROBLEM OF MEASUREMENT OF THE FINENESS OF. H. J. Henning. *Klepzig's Textil-Z.*, 1940, V. 43, P. 36–45; abs. in *C. A.*, 1940, V. 34, Col. 1854.

A detailed description of phys. methods of measurement; 12 references. (C)

V. Pure Science, Economics and Misc.

Some Thoughts on the Scientific Approach to Textile Problems. Albert C. Walker. *Rayon Text. Mo.*, June 1940, V. 21, P. 41–2; July 1940, V. 21, P. 45–6.

Statistical procedures which have been developed within recent years are an important form of scientific approach in controlling the causes of variation not only in a manufactured product but also in gauging the test method itself. The first step in analyzing data is to see if the test method is capable of giving reproducible results; that is, to determine if the results exhibit statistical control. Once this has been demonstrated it is possible to find out if the product is manufactured under technical control or whether some predominant cause of variation exists which may be found and corrected. (C)

Starches: Modified. Part III. G. B. Jambuserwala and K. R. Kanitkar. *J. T. I.*, Jan. 1940, P. T1–16.

In continuation of the work published as Parts I and II, the authors have degraded starches (corn, farina, sago, wheat) by means of (a) mixtures of potassium dichromate and sulphuric acid, using six different concentrations of the acid, the quantity of potassium dichromate, and therefore of available oxygen, being kept constant in all the six cases; (b) sulphuric acid alone of the same concentrations as in (a), and (c) alkaline hypochlorite solutions. Broadly speaking an exhaustive study of the properties of the modified starches produced in the present investigation has been made on the lines of Part II, and in addition determinations of the "binding values" of these products have been made. Further, the chemical and textile qualities of these products have been compared and correlated with some of the commercial "thin boiling" starches like Penetrose, Textiline and Fabricollin.

It is concluded that the present modified products compared very favorably with raw and commercial products with regard to their textile qualities such as (1) penetration, (2) increase in weight, (3) tensile strength, (4) feel (softer), and (5) binding values. Briefly, the degraded products are more adaptable to textile operations than the raw varieties. (C)

Starch: New Views on. Karl Freudenberg, Ernst Schaaf, Gertrud Dumpert and Theodor Ploetz. *Naturwiss.*, 1939, V. 27, P. 850–3; abs. in *C. A.*, 1940, V. 34, Col. 2632. (C)

The Stress-Strain Diagram of Isotropic Cellulose Fibres with Regard to the Theoretical Relations between Swelling, Anisotropy, Orientation, and Strength. P. H. Hermans. *Kolloid Z.*, December 1939, V. 89, No. 3, P. 344–8; abs. in *Eastman Kodak Abs. Bull.*, April 1940, P. 217.

The author shows that the degree of elongation of a stressed fibre in the dry state can be related to the stress-strain curves of diversely swollen fibres. In this manner the micellar orientation for a given elongation is assumed to be independent of the degree of swelling, and this suggests that the deformability of an isotropic fibre is predetermined by the nature of the sol. II. P. H. Hermans and P. Platzek. *Kolloid Z.*, December 1939, V. 89, No. 3, P. 349–54. Filaments prepared by different methods also showed that the micellar orientation can be related to the degree of elongation of the dry fibre in a manner which is independent of the degree of swelling. (S)